

# THE NOL RING TEST

## AN IMPROVED TOOL FOR CHARACTERISING THE MECHANICAL DEGRADATION OF NON-FAILED POLYETHYLENE PIPE HOUSE CONNECTIONS

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### ABSTRACT

Nowadays, determining the reliability of plastic pipe networks is a priority. In this context, inspired by the ASTM D2290-04 standard, a new test, called “Nol ring” (NR) has been developed by the Laboratoire National de Métrologie et d’Essais (LNE) in partnership with Suez-Environnement, to characterise the mechanical degradation of polyethylene house connection pipes. This new characterisation method was then implemented to 121 non-failed PE pipe samples excavated from the field.

For Suez-Environnement, the target was to define different degradation indexes to assess the overall, chemical and mechanical states of each sample. Different tests were performed. The Nol ring test was selected to assess the mechanical performance of PE pipes. Previous work detailed the Nol Ring classifications of degradation. Based on this definition, each sample was ranked according to the sample appearance and the elongation at break value. In addition a derivative index, called the NRR Index (Nol Ring Results), was defined including both of the above mentioned analytical parameters.

To classify the pipe degradation, three degradation levels were then defined and applied for each analytical parameter and/or degradation indexes:

- Green Level = no degradation observed and analytical parameter value in compliance with standards or requirements,
- Yellow Level = first signs of deterioration and analytical parameter value lower than the standards or requirements,
- Red Level = confirmed degradation and analytical parameter value in a critical range

The first results showed that the ranking with the NRR index is more discriminating than the initial NR categories of degradation. Furthermore, it appears that this three-degradation level approach used separately for the specimen appearance and the elongation at break value provides information regarding to the chemical oxidation and the material quality. Thus, the Nol Ring test and its degradation classifications are relevant for characterising the level of degradation of polyethylene house connection pipes and could also bring added value for the material selection and the quality control.

**KEYWORDS:** Nol ring, Polyethylene pipes, Characterisation, Degradation, Mechanical Performances.

## INTRODUCTION

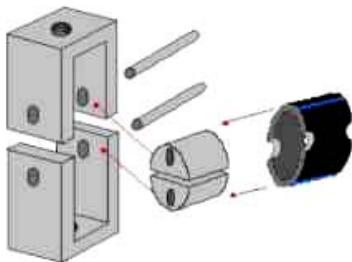
The development of polyethylene production processes and the increase of industrial production capacities in the fifties led to mass use of polyethylene (PE) for the distribution of gaseous fuel, drinking water and industrial applications. The success of polyethylene for the mentioned uses is linked to its advantages: low cost, set-up versatility, weldability, flexibility, density, chemical resistance and toxicological safety.

Some recent studies, Chung et al, Castagnetti et al, Colin et al [1-3], have shown that the most frequently used disinfectants, in particular chlorine, chloramines and chlorine dioxide, oxidise the inner surface of polyethylene pipes with a significant depletion of antioxidants. As a result, the PE becomes embrittled [4]. Micro-cracks appear and coalesce into large cracks which can propagate through the pipe wall and induce brittle failures, even after a very short time of use (especially for chlorine dioxide networks) relative to the service life of polyethylene pipes (which has to be over 50 years for conveying water at 20°C). Thus, the durability of these networks using common disinfectants becomes an economic and strategic parameter.

To assess the effect of disinfectants on polyethylene pipes, several kinds of data are required, such as precise pipe identification (classification, raw material, extrusion, date of production), its operating conditions (disinfectant, operational duration, background information) and the characterisation of its potential damage (internal or external).

In this context, it appears necessary to define the right testing to characterise the chemical and mechanical changes of polyethylene pipes, which have been exposed to common disinfectants used in water treatment.

For determining the mechanical performance of PE pipes, a new test called “Nol Ring”, has been developed by the Laboratoire National de Métrologie et d’Essais (LNE) with the partnership of Suez-Environnement. Inspired by the ASTM D2290-04 testing method [5], it was designed to be simple to carry out and cheaper than the conventional hydrostatic pressure test. Figures 1 and 2 illustrate the apparatus scheme and a typical brittle failure observed in the cut section.



**Figure 1: Testing Apparatus for Nol Ring measurement**



**Figure 2: Failure occurred in the cut section**

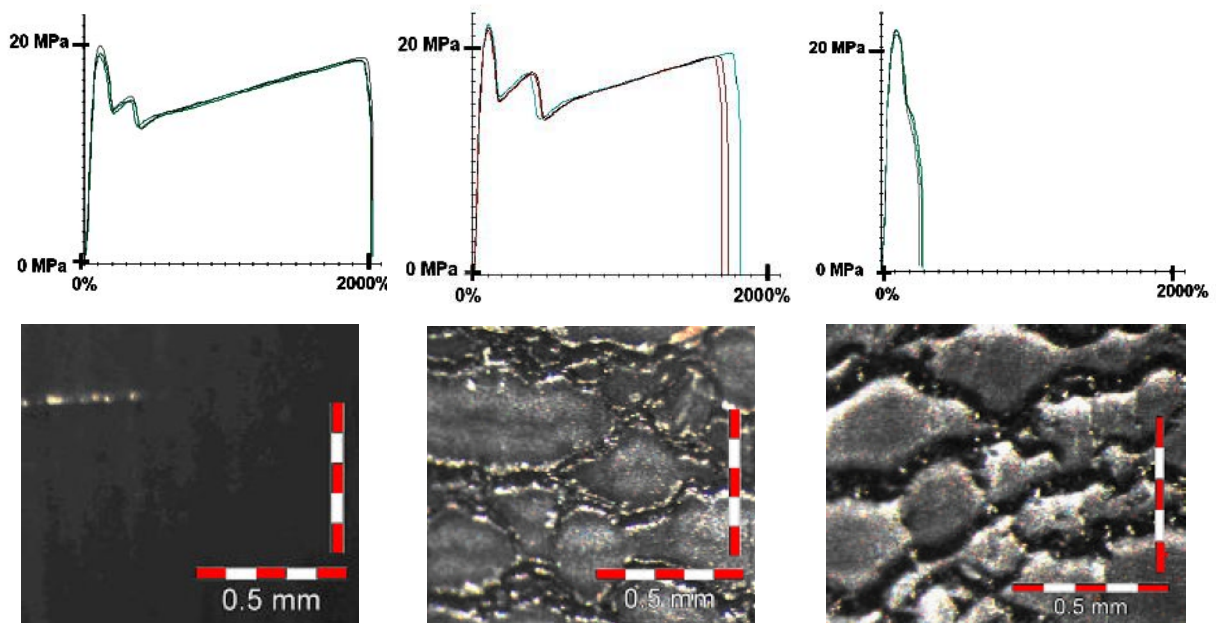
Previous work [6] showed that this developed test, compared to the quick burst test, induces a hoop (circumferential) stress, which is quite similar to the stress induced by internal pressure. The results demonstrated that the Nol ring test is thus more relevant for identifying pipe damages than the standard tensile test according to ISO 6259-1/3. Further severe and unexpected pipe degradation can be revealed. With this test, two main characteristics were found to be relevant to assessing the degradation of the pipe:

- The surface aspect of the specimen’s inner wall after the test (viewed without magnification)
- The elongation at break

Based on these two characteristics, obtained from the Nol ring tensile curves, five different degradation levels were identified as described below:

- Level [0] - No modification of the surface aspect during the test. The break starts from the inner side of one stressed area. The elongation at break is high and always  $\geq 1500\%$ . The stress at break is always higher than the stress at the second yield point.
- Level [1] - A light bleaching appears and sometimes a weak roughness can be observed. The other characteristics are the same as [0].
- Level [2] - Bleaching, scores and roughness are observed on the surface. The break starts from the inner wall of the pipe, on one stressed area. The elongation at break is generally between 1100% and 1500%. The stress at break is often higher than the stress at the second yield point.
- Level [3] - Bleaching, scores and roughness on the surface are obvious. The break starts from the inner wall of the pipe and can be located between the two edges of the stressed areas. The elongation at break is low: between 150% and 1100%. The stress at break is often lower than the stress at the second yield point.
- Level [4] - A white line appears between the two stressed areas on the inner wall of the pipe. The failure is initiated from this line and the elongation at break is low. The second yield point is not observed.

For instance, Figure 3 illustrates the obtained results for 3 of these classified degradation levels.



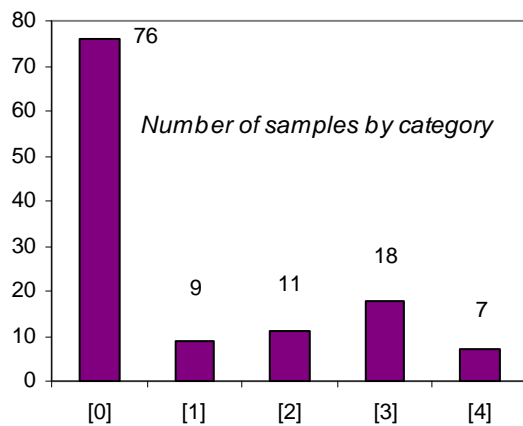
**Figure 3: Nol Ring Tensile Curves and characteristic surface aspect of the specimen inner wall after testing: degradation levels [0], [2] and [4] from left to right**

The brittleness is revealed by a low value of the elongation at break corresponding to scoring and roughness of the inner pipe wall between the stressed areas after testing.

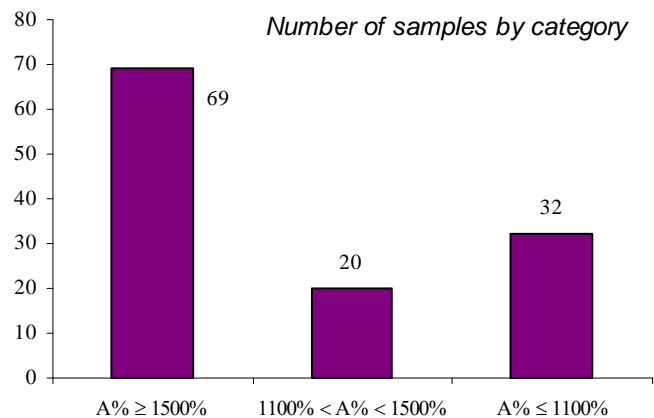
At this stage, the testing methodology needed to be implemented on several field samples for validation.

## IMPLEMENTATION OF NOL RING TEST ON FIELD SAMPLES

Within the scope of a 3-year R&D program, Suez-Environnement led a large sampling campaign to collect PE pipe samples, representative of the current buried PE pipe networks. Several criteria were targeted to select the collection sites: the disinfectant nature and concentration, the water quality, the water temperature, the pressure, the age or the PE grade. A total of 201 non-failed and 91 failed samples were excavated in 7 different countries: France, Italy, the UK, Spain, the USA, Morocco and Hungary. Among them, 121 samples were analysed by the Nol ring test and ranked based on the defined NR degradation categories. Figures 4 and 5 respectively show the resulting ranking according to both of Nol Ring's degradation parameters, i.e. the specimen appearance and the elongation at break value.



**Figure 4. Nol Ring test results according to specimen appearance**



**Figure 5. Nol Ring test results according to the Elongation at break value A%**

However, some samples exhibited contradictory results between the surface aspect of the sample inner wall and the elongation at break value and needed additional investigation.

## CHARACTERISATION OF PE PIPE DEGRADATION

As the degradation could be the result of chemical and/or mechanical damage, 5 degradation indexes were defined to distinctly characterise the chemical, overall and mechanical degradation of the analysed PE samples.

### Chemical ageing:

- Oxidation Induction Time (OIT): measures the residual content of antioxidants.
- Carbonyl Index (CI): quantifies the formation of oxidation products.

### Overall embrittlement of the inner pipe wall:

- ODL Index (“Overall Degradation Layer”).

This new index results from examining the inner pipe wall surface by optical microscopy before and after the reverse bend back test (RBBT).

Reverse Bend testing was achieved in accordance with ASTM D2513-06 Section A1.5.11.1 with the exception of test temperature and specimen test width. A 13mm wide ring was cut from each pipe sample with outer diameter ≤ 50 mm. For those ones with outer diameter >50mm, a 25mm wide ring was taken.

An arc with the width of at least  $\frac{3}{4}$  of the total ring was cut from the ring and bent back to examine for:

- Whitening of the pipe's inner wall;
- Crazing: formation of low-density longitudinal entities typically accompanied by fibril materials oriented along a short axis;
- Micro-cracking: formation of fine cracks that are not visible to the naked eye but may be observed easily under a microscope or SEM;
- Cracking: formation of cracks visible to the naked eye or brittle failure of the sample during the bending.

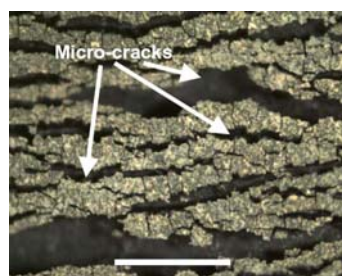
Cracking and micro-cracking can occur together, as may micro-cracking and crazing, as illustrated by the microscopic observations in Figures 6, 7 and 8.



**Figure 6: Cracks in sample surface prior to RBBT**

Field sample N°1 (PE32)

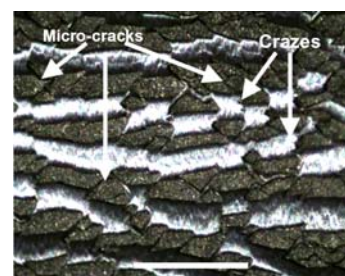
Scale: bar = 1mm



**Figure 7: Typical micro-cracking after RBBT**

Field sample N°1 (PE32).

Scale: bar = 1mm



**Figure 8: Typical micro-cracking and crazing after RBBT**

Field sample N°2 (PE32).

Scale: bar = 1mm

The specimen cross-section is also examined to look for flaws or micro-cracks in the pipe wall. Based on the observations of nearly 300 samples, 8 degradation levels were identified and ranked according to the ODL index.

#### Mechanical performance:

- NRR Index: obtained from the Nol ring test

As the specimen appearance and the elongation at break value are complementary criteria, a derivative index, called the NRR Index (“Nol Ring Results”) integrates both analytical parameters.

- HI index: refers to the sustained hydrostatic pressure test performed at 90°C in compliance with ISO 1167:1996 and ASTM D1598-02 as a screening test for evaluating pipe performance in end-use conditions.

Next, to easily classify each analysed sample into an appropriate degradation category, a three-level approach was adopted for each newly defined index with the following definition:

- Green Level = no degradation observed and analytical parameter value in compliance with standards or requirements,
- Yellow Level = first signs of deterioration and analytical parameter value lower than the standards or requirements,
- Red Level = confirmed degradation and analytical parameter value in a critical range

Table 1 shows the newly defined classification for each degradation index.

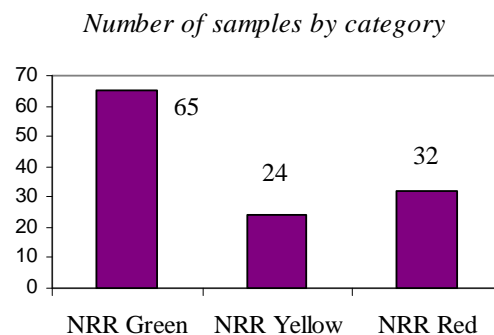
**Table 1: Characterisation of the PE pipe degradation into three levels by 5 degradation indexes**

<b>Degradation category</b>	<b>Green Level</b> <i>No degradation</i>	<b>Yellow Level</b> <i>Moderate degradation</i>	<b>Red Level</b> <i>High degradation</i>
<b>OIT</b> <i>Chemical ageing</i>	OIT > 20 min	5 min < OIT ≤ 20 min	OIT ≤ 5 min
<b>CI Index</b> <i>Chemical ageing</i>	CI < 0,05	0,05 < CI ≤ 0,1	CI ≥ 0,1
<b>ODL Index</b> <i>Overall embrittlement</i>	(8) = no crazes (7) = limited crazing	(6) = Moderate crazing (5) = extensive crazing	(4) = limited micro-cracking (3) = Moderate micro-cracking (2) = extensive micro-cracking (1) = brittle failure
<b>NRR Index</b> <i>Mechanical performance</i>	[Appearance] < 2 <b>AND</b> A% > 1500%	[Appearance] = 2 <b>OR</b> 1100% < A% ≤ 1500%	[Appearance] > 2 <b>OR</b> A% ≤ 1100%
Specimen Appearance	[0] or [1]	[2]	[3] or [4]
Elongation at break value A%	A% > 1500%	1100% < A% ≤ 1500%	A% ≤ 1100%
<b>HI Index</b> <i>Hydrostatic Pressure resistance</i>	> 672 hours	336 h < HI ≤ 672 h	HI ≤ 336 h

Integrating this new methodology, the pipe degradation assessment was performed for all the analysed samples, and the reliability of each index compared.

### COMPARISON BETWEEN THE NOL RING TEST RESULTS AND THE OTHER DEGRADATION INDEXES

With this new approach, Figure 9 shows that the classification with the NRR index is more severe than those obtained with the first defined NR degradation categories. Indeed, 20 samples were differently classified (13 samples moved into the Yellow level and 7 other ones into the Red category).



**Figure 9: Sample ranking according to the NRR index**

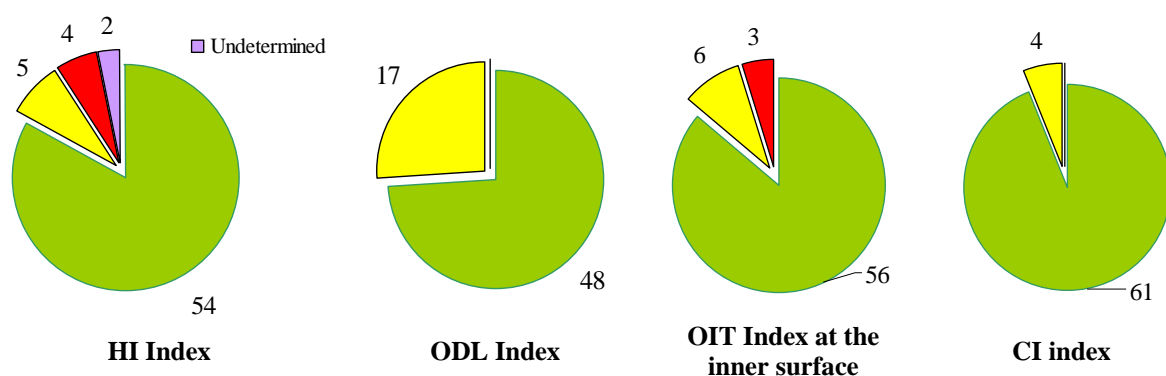
However, it was still noticed that, for a same sample, the ranking by degradation level (“Green, Yellow, Red”) could differ between the specimen appearance or the elongation at break value, as shown in Table 5.

**Table 5: Degradation categories observed for a same sample according to the specimen appearance, the elongation at break value and the associated NRR index**

Parameter	Specimen Appearance	Elongation at break value A%	NRR index
<b>Degradation category</b>	Green	Green	<b>Green</b>
	Yellow <sup>(a)</sup>	Yellow <sup>(a)</sup>	<b>Yellow</b>
	Yellow <sup>(b)</sup>	Green <sup>(b)</sup>	
	Green <sup>(c)</sup>	Yellow <sup>(c)</sup>	
	Red <sup>(a)</sup>	Red <sup>(a)</sup>	<b>Red</b>
	Red <sup>(b)</sup>	Yellow <sup>(b)</sup>	
	Green <sup>(c)</sup>	Red <sup>(c)</sup>	

Further investigations were performed for each NRR Index category (Figure 9), taking into account the operating conditions and the disinfection process. Next, the NRR Index was compared to the results obtained from the other degradation indexes.

For the Green Degradation Level (NRR Green), no clear differences were noticed between the disinfection processes: 46 samples were exposed to chlorine, 15 to chlorine dioxide, 1 to monochloramines and 3 without any disinfection. Figure 10 illustrates a good correlation between the NRR Index and other indexes. The majority of the samples do not exhibit any oxidation and maintain their mechanical performance.



**Figure 10: Comparison between “Green” NRR index and other degradation indexes**

A similar connection to the other degradation indexes was observed for the highest degradation category, corresponding to a “Red/Red”<sup>(a)</sup> NRR Index (See Figure 11). Indeed, for those samples, the consumption of antioxidant was complete at the inner wall, combined with a high oxidation level leading to an embrittlement of the inner surface. In addition, about 2/3 of the samples showed a poor hydrostatic HI index, i.e. poor pressure resistance.

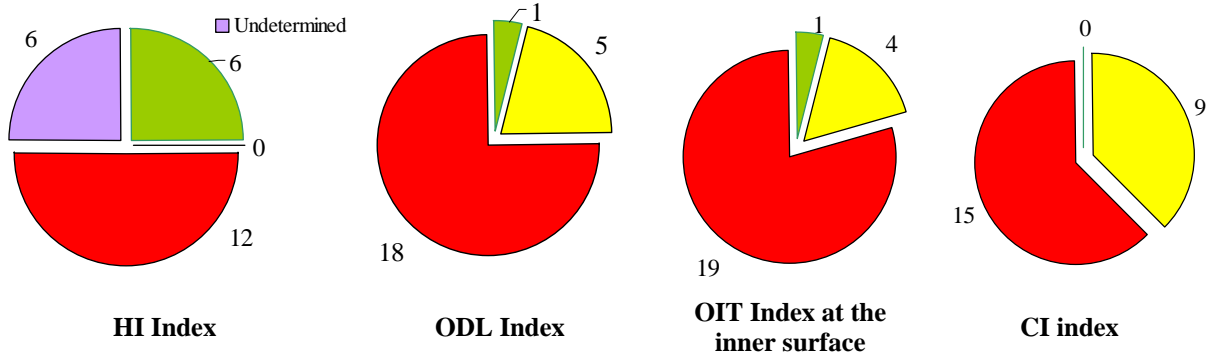
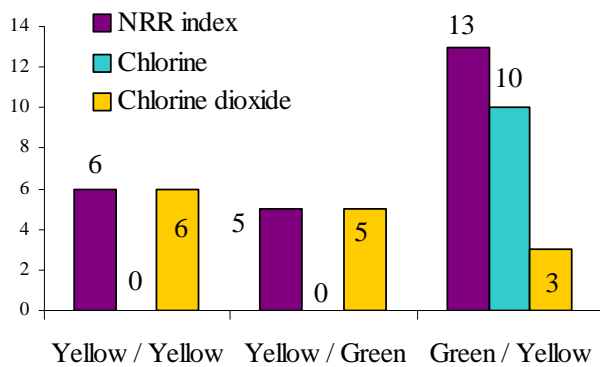


Figure 11: Comparison between the "Red/Red" NRR index and other degradation indexes

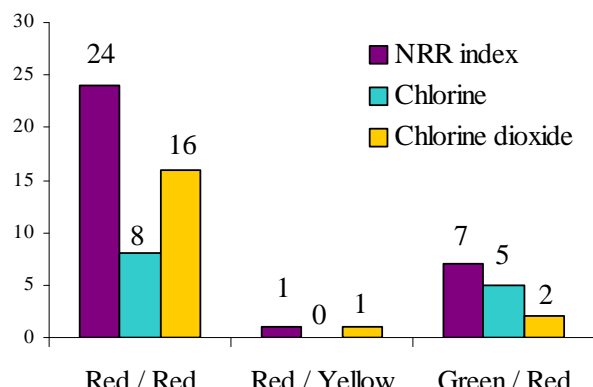
Regarding to the disinfection process (See Figures 12 and 13), the Red/Red NRR Category corresponds mainly to PE pipes exposed to chlorine dioxide-disinfected water. The remaining 8 samples with chlorine correspond to the oldest specimens (30 years old) in Northern Africa and Southern Europe, where water temperature is high. For the yellow/yellow NRR class, all 6 of the samples were also disinfected by chlorine dioxide.

Therefore, these 30 samples<sup>(a)</sup>, which constitute nearly 25% of the analysed samples, showed a moderate or high level of mechanical degradation combined with a moderate or high oxidation level, respectively, at the inner pipe wall.



Yellow NRR Index: [Aspect] / A% classification

Figure 12: Moderated Degradation Level  
Distribution according to disinfectant type



Red NRR Index: [Aspect] & A% Classification

Figure 13: High Degradation Level  
Distribution according to disinfectant type

Furthermore, two additional groups<sup>(b,c)</sup> of samples could be noticed according to the NR analytical parameters, as detailed in Table 5:

- "Yellow" specimen appearance/"Green" A% value, or a "Red"/"Yellow" combination, respectively(b): 6 samples were identified, as illustrated in Figures 12 and 13.
- "Green" specimen appearance combined with a "Red" or "Yellow" elongation at break value(c). This combination is exhibited by 20 samples.

Figures 14 and 15 detail the results obtained with the other degradation indexes for both classes of samples.

Firstly, Figure 14 demonstrates that all these samples are chemically oxidised. The depletion of antioxidants was focused on the inner wall, without any consumption in the mid-wall and outer wall. The presence of oxidation products and an embrittlement of the inner pipe wall were also noticed. Thus, this category of samples maintains a good or a moderate mechanical resistance even if the chemical oxidation process has already started.

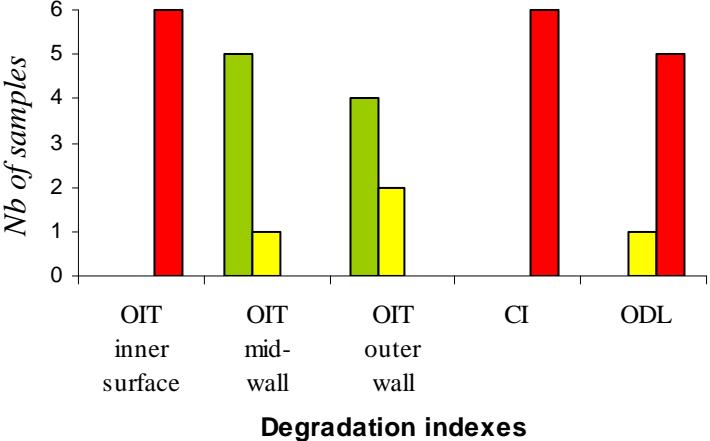


Figure 14: Yellow/Green and Red/Yellow classes for Nol Ring [Aspect] and A% value

At the opposite end, Figure 15 clearly indicates another phenomenon for pipe degradation. In this case, the “Green” level classification of all OIT, CI and ODL indexes refers to poor quality materials rather than samples oxidised by chemical ageing.

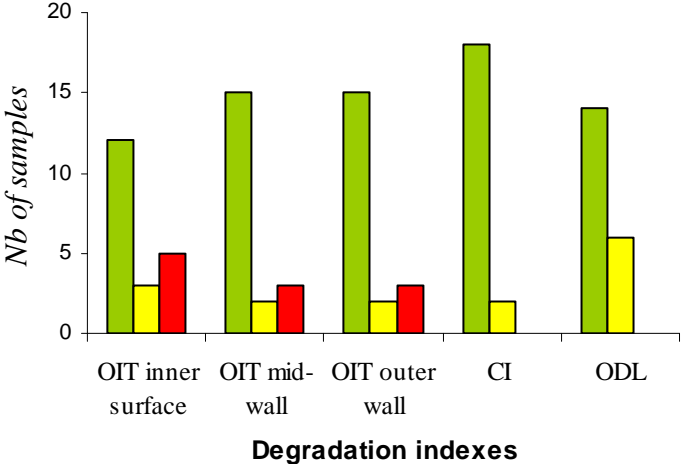


Figure 15: Green/Yellow and Green/Red classes for Nol Ring [Aspect] and A% value

Consequently, the NRR index and its associated analytical parameters, which are the appearance of the sample surface and the elongation at break after testing, make it possible to characterise the mechanical performance of PE pipes under operating conditions. Furthermore, combined with other degradation indexes, the origin of the degradation can be assessed.

## CONCLUSION

To classify PE pipe degradation, different tests were performed to assess the overall, chemical and mechanical states of each sample. Amongst them, the remaining mechanical performance of PE pipes was determined by the Nol Ring (NR) test. For quick and easy categorisation, a three-degradation level approach was defined as follows: “Green” = No degradation, “Yellow” = Moderate degradation, “Red” = High degradation. They were applied to 5 degradation indexes and their analytical parameters. For Nol Ring test results, the NRR index takes into account the surface appearance and the elongation at break value after testing.

With this new approach, the classification with the NRR index appeared more discriminating than the previous one and allowed a good preliminary ranking of the samples. However, differences were noticed for the same NRR Index that led to detailing the three-degradation level approach to the specimen appearance and the elongation at break value and comparing the Nol ring results to those obtained from other degradation Indexes. The results were successful with the identification of 4 main classes of samples:

- No chemical oxidation and good mechanical performance
- The chemical ageing had begun but with no impact on mechanical performance
- Strong chemical oxidation correlated with poor mechanical performance
- No chemical oxidation but poor mechanical resistance. This last category refers to poor quality materials rather than samples oxidised by chemical ageing.

Therefore, the Nol Ring test, combined with other degradation indexes, is relevant for characterising the level of degradation of polyethylene house connection pipes under usual operating conditions and could also bring added value for the processes of material selection and quality control.

## REFERENCES

- [1] S. Chung, K. Oliphant, P. Vibien and J. Zhang, *An examination of the relative impact of common potable water disinfectants (Chlorine, Chloramines and Chlorine dioxide) on plastic piping system components*, in Proc of ANTEC, 2007, **5**, 2894 – 2898.
- [2] S. Chung, T. Li, K. Oliphant, P. Vibien, M. Rozental-Evesque and B. Rabaud, *Characterizing long-term performance of plastic piping materials in potable water applications*, in Proc of PPXIV Conference, 2008, October 22 – 24, Budapest, Hungary.
- [3] D. Castagnetti, E. Dragoni, G. Scirè Mammamo, N. Fontani, I. Nuccini and V. Sartory, *Effect of chlorinated water on the oxidative resistance and the mechanical strength of polyethylene pipes*, In Proc. of Plastic Pipes XIII, 2006, October 02 – 05, Washington, DC, USA.
- [4] X. Colin, L. Audouin, J. Verdu, M. Rozental-Evesque, F. Martin and F. Bourguine, *Kinetic modeling of the aging of polyethylene pipes for the transport of water containing disinfectants*. In Proc. of the plastic pipes XIII conference, 2006, October 02 – 05, Washington DC, USA.
- [5] ASTM D 2290-04: 2000, *Standard Test Method for Apparent Hoop Tensile Strength of Plastic or Reinforced Plastic Pipe by Split Disk Method*.
- [6] M. Sanchez, S. Louis, C-E Bruzek, M. Rozental-Evesque, B. Rabaud and K. Glucina, *Development of a “Nol ring” test to study polyethylene pipe degradation*, in Proc. of PPXIV conference, 2008, October 22 – 24, Budapest, Hungary.