

UV Test Method Comparison

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Introduction

This report is based on facts and experiences of

- PESTEC test results of WOM, XENO and SEPAP tests
- Olivier Haillant Weathering Expert Consultant of ATLAS Company (biggest Manufacturer of all kind of UV testing equipments since 1915 – also including WOM, XENO and SEPAP test equipments
- Joachim Kohler Technical Service Clariant Company one of the biggest additive suppliers worldwide

<u>Target</u>

Target of UV test of stay cable pipes is the verification to comply with the durability requirements for stay cable bridges.

The characteristic for the test program should be

- accelerated
- realistic
- representative same chemical formulation
- precise / reproducibility
- global

Polymer Oxidation and UV Light properties

Light, heat and shear are initiators which lead to the oxidation of polymers by air. The rate of this process depends on the type of polymer, its quality and stabilization.

The most harmful part of the natural sun is the UV light. Although its share is just about 6.8 %, UV is more or less responsible for 100 % degradation of organic materials, in particular polymers.

The shortest wave length of sun-light reaching the earth's surface by passing the atmosphere is about 290 nm (shortwave UV light). To simulate sun light by artificial weathering devices, two main aspects need to be considered.



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First is the **range of UV irradiation (290 – 400 nm)** and second the **Spectral Energy Distribution** has to be as close as possible to natural UV.

Sunlight vs. Artificial Light sources

Comparison of Spectral Energy Distribution in different artificial weathering devices



Green = Xenon-Arc Lamp (Xenon/WOM Test), Red = Mercury-Arc Lamp (SEPAP Test)

Conclusion for natural uv-light in the range of 290-400 mm.

Xenon-Arc is very close

Mercury-Arc is quite different

Therefore most international standards for accelerated weathering are based on Xenon Arc irradiation.

Mercury irradiation is known in the market since decades. Although its importance as artificial weathering device is low, it still is applied as it has the capability to induce very fast photodegradation caused by the extraordinary high energy level.



XENO / WOM Test - General

Accelerating **weathering Lab Test** using Xenon Arc light. Specimen are continues exposed to radiation in 102/18 minutes dry/rain cycles at humidity of 60-80% according ASTM G 155 Cycle 1 or ISO 4892.2 Cycle 1 norms.

Result evaluation is either made by optical comparison of specimen according Greyscale ISO 105 A02 or by colorimetric measurement acc. ASTM E 1347 or ISO 7724-3 with results called deltaE. WOM tests are used in all industries.

PESTEC Test duration 20000 hrs = 2,4 years to get results equivalent to 20 years outdoor exposure. Also 1500 - 4000 hrs = 2-6 month test equivalent to 1,5 - 4 years exposure will provide first expectations.

WOM Test equipments are available all over the world at many different test laboratories.

WOM Test – Details

Several colormetric measurement systems have been developed in the past. Today the most popular is the Cie-Lab system, based on the measurement of the light incidence of a wavelength for each color. The results are displayed in a three-dimensional color system, which enables the user to express each color in a numeric value and so ensures its reproducibility.

Each color is clearly defined by three coordinates, named L*, a* and b*.

- L* = light/dark value (axis)
- a* = red/green value (axis)
- b* = yellow/blue value (axis)



Color comparison results are based on the calculated differences between the coordinates in the 3-dimensional system. The reference value is called E*Lab.

To compare a determined color (here called "Y") versus the reference color (here called "R") both values are calculated and the result is value ΔE^*Lab .



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6.03.03 Equation: CIE Lab		Optiview Quality Control Version 5.1.3.0226				15:40		
CE-7000A - XA0761		Status: CRIOSS			Illuminant: D65			
STANDARD: Pipe Sample af	ter Production		otatus, orta			MEASUREM	ENT TYPE: Re	eflectance
Standard	Illum	L*	a*	b*				
Pipe Sample after Production	D65	40.483	41,473	23.035				
	A	46.633	42.909	33,701				
	TL84	44.080	41.637	29.158				
Trial	Illum	L*	a*	b*	DI *	Da*	Db*	DE*
N0 1, 11 years south europe	D65	37.128	36.913	19,164	-3.356	-4 560	-3.871	6 850
	A	42.561	38.520	28.618	-4.072	-4 389	-5.083	7 854
	TL84	40.269	37.263	24.491	-3.811	-4.373	-4 667	7 445
No 1., 11 years south europe	D65	39.258	40.681	24.308	-1.225	-0.792	1 273	1 936
	A	45.319	41.636	34.780	-1.314	-1.273	1.080	2 124
	TL84	42.955	41.268	30.635	-1.125	-0.368	1 477	1 893
No 2., 11 years south europe	D65	37.338	37.050	19.574	-3.145	-4.423	-3.462	6 437
	A	42.803	38.637	29.069	-3.830	-4.272	-4.631	7 374
	TL84	40.501	37.414	24.943	-3.579	-4.223	-4.215	6.958
No 2., 11 years south europe	D65	39.321	40.657	24.313	-1.163	-0.816	1.277	1 910
	A	45.381	41.646	34.780	-1.252	-1.263	1.080	2 080
	TL84	42.998	41.210	30.608	-1.082	-0.427	1.449	1 858



Multitrial Color Difference/Spectral Graph Report

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SEPAP 12.24 Test - General

Accelerating **photo-aging Lab Test** using Mercury Arc light. Specimen are continues exposed to monochromatic radiation. SEPAP is only referred to according an AFNOR standard for greenhouse and silage films, extended to European standard in 2002This is no weathering test and does not simulate sunlight.

Result evaluation is made by IR analysis <u>(FTIR/IRTF)</u> of specimen microtome cuts 50-100 microns by screening the C=O value (carbonyl Index).

Test duration 1500-4000 hrs = 2-6 month equivalent to 2,5 - 7 years exposure SEPAP Test equipments are available only in France and experiences in result evaluation are with Prof. Lemeire from CNEP lab only.

SEPAP 12.24 Test – Details

Despite the differences in radiation energy in the uv-spectrum, the IR Analysis method is a fast method to identify virgin polymer degradation in most of the tests and in many cases the value is proportional to the real polymer degradation.

Even some polymer batches including pigments and stabilizers might be evaluated, in particular altered polymer batches often cause non-realistic degradation, since the C=O rate of the polymer degradation is higher and overlay the additives, so they will not be detected. Also some pigments (e.g. organic red colour) and additives can absorb UV light between 270 and 300 nm which provokes their decomposition after the activation energy was overcome.

Additive migration depends on the material wall thickness and the thickness of oxidized layer (TOL) which varies with the chemical formula of each polymer.

SEPAP simplify by evaluating only 50-100 μ m of total 2.000 μ m colored pipe wall thickness additional average black wall thickness of 3.500 – 10.000 μ m and so does not reflect the overall pipe stabilization due to the different additive migrations.



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SEPAP Test Specimen

Following effects might falsify the evaluation results.

- use of non-natural light radiation with very high energy causing molecule absorptions, polymer and additive damages and excessive polymer oxidation
- not all additives, UV stabilizers, antioxidants and pigments are detectable with this method
- some additives contain C=O groups leading to a C=O index right from the beginning
- + 50-100 μm microtome evaluation only
- IR evaluation method not generally suitable to detect all kind of polymers and additives
- Different test results by comparison of acceleration tests

Prof. Lemaire from CNEP laboratory invented the SEPAP 12-24 test about 20 years ago originally for testing of thin films (up to $50 \ \mu$ m).

The SEPAP Test did not reach international acceptance up to now and has not been approved from experts for an international norm.



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PESTEC NOCX Pipe Tests

PESTEC NOCX pipes with 7 different colours in 4000 hrs SEPAP 12.24 test varied in result from good for colour "light grey" to completely determine for colour "red" after test time of already 200 hrs. (6 month) versus same PESTEC NOCX pipes in 20000 hrs Xenotest 1200 and W-O-M Test (corresponding to 20 years exposure) show very good uv-stabilization results for all colours.

Same red pipe has been installed 15 years ago on a bridge in South Spain and the color and surface still remain in very good conditions.

In both comparisons SEPAP does give realistic prediction of long term polymer stabilization. Also SEPAP did not detect our uv-stabilizers (HALS), obviously caused by one of the above mentioned test method falsification.

Conclusion

PESTEC propose to refer to standardized WOM Test acc. international accepted ISO and ASTM norms as a reference test for prediction of HDPE cable stay pipe durability.

Specification proposal:

- Xenon Arc Light weathering test according ASTM G 155 Cycle 1 or similar ISO 4892.2 for 1.500 hours. Results of minimum grade 4 according ISO 105 A02 greyscale or result of max 3.0 Delta E lab according Colorimetric measurement ASTM E 1347 (or similar ISO 7724-3). Results to be examined once for each color.
- Mechanical property Test of 1500 hrs tested specimen to reach yield stress of min 16 Mpa and elongation at break min 300%.
- Project Engineer might consider approving existing test results of colors similar with the specified project color.