# INTERNATIONAL STANDARD



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# Plastics — Artificial weathering including acidic deposition

Plastiques — Vieillissement artificiel y compris les dépôts acides

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<u>ISO 29664:2010</u> https://standards.iteh.ai/catalog/standards/sist/ce7d8cc9-4f63-4476-9691-534afd512eac/iso-29664-2010



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### Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 29664 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

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### Introduction

This International Standard specifies methods to reproduce the weathering effects that occur when plastics are exposed to heavily polluted outdoor environments. Acidic precipitation can have a significant effect on the photochemical ageing of many polymers. In some cases, this is due to attack on the stabilizers<sup>[5][6]</sup> or fillers like  $CaCO_3^{[7][8]}$  applied in technical polymers. The mechanisms differ from those of harmful gases, which essentially constitute the initial products of acidic precipitation.

Arising from changing industrial air pollution and additionally diffused by the stochastic wind and cloud distribution, acidic precipitation occurs sporadically. Thus, especially regarding acidic precipitation, outdoor weathering effects vary enormously within different years. Therefore, it is practically impossible to obtain reliable outdoor exposure results from just one season. These fluctuations can be avoided through the use of a laboratory test, where all weathering parameters, including the acidic deposition, can be controlled.

In artificial weathering tests for plastics, the action of acidic atmospheric precipitation, which is part of the real world, has generally not been considered.

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# Plastics — Artificial weathering including acidic deposition

### 1 Scope

This International Standard describes artificial weathering tests intended to evaluate plastics for use in heavily polluted outdoor environments. Results from this International Standard cannot be used to predict the service life of these plastics.

This International Standard describes two different exposure methods. Use of the methods depends on the form of the plastic product being evaluated.

Method A is intended for products where surface degradation is very important and uses a strong acid spray (pH 1,5) that is applied for a short time.

Method B uses a weaker acid spray (pH 3,5) that is applied over a long period of time so that it can penetrate deeply into the product and is intended for products such as geotextiles and related products.

This International Standard does not cover the influence of special chemicals like agrochemicals.

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# 2 Normative references (standards.iteh.ai)

The following referenced documents are indispensable for the application of this document. For dated references, only the references plies geordundated references, 4thé-9atest edition of the referenced document (including any amendments) applies eac/iso-29664-2010

ISO 291, Plastics — Standard atmospheres for conditioning and testing

ISO 4582, Plastics — Determination of changes in colour and variations in properties after exposure to daylight under glass, natural weathering or laboratory light sources

ISO 4892-1, Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance

ISO 4892-2, Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps

ISO 4892-3, Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps

### 3 Principle

The combined action of UV radiation, heat, humidity, wetting, and acidic deposition is evaluated in weathering devices conforming to ISO 4892-1, ISO 4892-2 and ISO 4892-3.

The acidic deposition, in some cases, may accelerate the degradation of the polymer itself. In other cases, the degradation is accelerated when the acid deposition attacks the stabilizers in the polymer.

Two different methods combine acidic deposition with artificial weathering. In method A, a strong acid solution is applied once per day. In method B, a weaker acid exposure is integrated in the rain phase.

Both test methods aim for the same physical-chemical degradation mechanism as known to occur from exposure in heavily polluted outdoor environment. To consider different climatic conditions, method A includes

two procedures, one intended to simulate very humid climates such as those in tropical or subtropical areas and one intended to simulate more moderate, less humid climates. As this mechanism does not depend on the size of the damage spots, either horizontal or vertical specimen orientation is allowed (see A.3).

Use of reference materials is recommended in order to monitor the consistency and repeatability of the test. Suited sets of reference materials may be samples with different grades (good, moderate, low performance) based on previous tests using the same method. The reference material selected shall be stable when stored in the dark at room temperature.

### 4 Apparatus

**4.1 General apparatus**, including two types of weathering devices conforming to ISO 4892-2 and ISO 4892-3. The test chambers, as well as all required mounting devices and holders, shall be made of acid- and UV-resistant materials.

NOTE As both types of weathering devices show a different spectral distribution, the results can differ.

**4.2** Artificial acidic deposition, to produce an acid mixture of pH 1,5 at  $(25 \pm 2)$  °C, add 10,6 g of H<sub>2</sub>SO<sub>4</sub>, 3,18 g of HNO<sub>3</sub> and 1,80 g of HCl to 10 litres of deionized or distilled water. This yields a mass ratio of 1,0:0,3:0,17. The quantity of acid to be added assumes that the concentration is 100 %. Since the actual concentration of the reagent grade acids used is less than 100 %, the amount of each acid added should be adjusted for the actual percent acid in the reagent. For example, if the concentration of the reagent HCl is 36 %, the actual amount of the reagent to be added is 1,8 divided by 0,36 or 5,0 g.

If required, a solution with a pH of 2,5 can be produced by diluting the pH 1,5 solution with demineralized water in a volume ratio of 1:10. (standards.iteh.ai)

The solution with a pH of 3,5 is prepared by diluting the pH 1,5 solution with demineralized water in a volume ratio of 1:100. ISO 29664:2010

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Check the pH of the acid solution before starting the test. If the pH is more than 0,3 units away from the desired pH, discard the solution and make a fresh batch.

Store the acid solution in a sealed container to avoid changes in pH during storage.

#### 4.3 Laboratory radiation sources.

**4.3.1 Fluorescent UV lamp apparatus**, conforming to ISO 4892-3 with Type 1A lamps specified in ISO 4892-3, if used.

**4.3.2** Xenon arc lamp apparatus, conforming to ISO 4892-2, equipped with daylight filters as specified in ISO 4892-2, if xenon arc apparatus is used.

Irradiance uniformity shall be in accordance with the requirements specified in ISO 4892-1.

**4.3.3 Irradiance**, UV irradiance of either  $0,76 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$  at 340 nm or 45 W·m<sup>-2</sup> in the 290 nm to 400 nm bandpass for fluorescent UV lamp apparatus operating with Type 1A lamps. During equilibrium operation, the allowed deviation of the measured irradiance shall be  $\pm 0,02 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$  when controlling at 340 nm and  $\pm 1,2 \text{ W}\cdot\text{m}^{-2}$  when controlled in the 290 nm to 400 nm bandpass.

For xenon arc lamp apparatus, operating with daylight filters, the UV irradiance shall be either 0,51 W·m<sup>-2</sup>nm<sup>-1</sup> at 340 nm or 60 W·m<sup>-2</sup> in the 300 nm to 400 nm bandpass. During equilibrium operation, the allowed deviation of the measured irradiance shall be  $\pm 0,02$  W·m<sup>-2</sup>nm<sup>-1</sup> when controlling at 340 nm and  $\pm 1,2$  W·m<sup>-2</sup> when controlled in the 300 nm to 400 nm bandpass.

If the indicated irradiance is outside the allowed deviations at equilibrium conditions, stop the test and determine the cause of the problem before continuing.

NOTE The irradiance limits given above apply only to the position where the irradiance is normally measured. Irradiance can vary by about  $\pm 10$  % across the whole specimen area.

**4.4 Test chamber**, of variable design, but constructed from inert material and conforming to ISO 4892-1 and ISO 4892-2 for xenon arc lamp devices and ISO 4892-3 for fluorescent UV lamp devices. The test chamber shall provide for control of irradiance, chamber temperature, and in case of a xenon arc lamp apparatus of black standard temperature (BST). The test chamber shall also provide for humidity control that meets the requirements of ISO 4892-1, if required. Devices shall also provide for water spray.

**4.5 Radiometer**, complying with the requirements outlined in ISO 4892-1, if used.

**4.6** Black standard thermometer, complying with the requirements for these devices which are given in ISO 4892-1.

#### 4.7 Wetting and humidity

#### 4.7.1 General

Specimens shall be exposed to moisture in the form of relative humidity (RH) during the dry periods and in the form of water spray during the wet periods.

**4.7.2 Relative humidity control equipment**, for use in controlling the relative humidity during the dry periods, with the location of sensors used to measure humidity in accordance with ISO 4892-1.

NOTE 1 Generally, the level of relative air humidity has a significant influence on photodegradation of many polymers.

NOTE 2 Additionally, relative air humidity influences the effect of the acidic precipitation. The several acids and water have different vapour pressures and, therefore, evaporate at different rates depending on the temperature and especially on the relative humidity. The acid concentration of the solution gradually increases, from about 0,01 % acid after spraying to about 60 % H<sub>2</sub>SO<sub>4</sub>.

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To ensure repeatable evaporation processes and get a slow change of the composition of the acids to enable each acid type enough and reproducible impact duration, relative humidity should be controlled. Particularly for aqueous sulfuric acid solutions, relative humidity interacts with the acid concentration (see ISO 483). That means, by controlling the relative humidity in the weathering device, the acid concentration of the aqueous acid solution can be controlled between 5 % and 70 %.

**4.7.3** Water spray system, capable of introducing intermittent spray of high purity water to the exposed surface of the specimen in test chambers used for method A. Test chambers used for method B shall be equipped with means to introduce intermittent spray of an acidic solution with a pH of 3,5 to the exposed surface of the specimen. In all cases, the spray shall be uniformly distributed over the test specimens. The spray system shall be made from corrosion-resistant materials that do not contaminate the water or acid solution used.

If the water is recycled, the quantity of circulating water should not be less than 50 litres.

If recycling should be used for the spraying water, the water should be replaced regularly and at least once per week of operation (168 h). Because of the uptake of the acids washed off the specimens and possible contamination from the sample's material, for method A, the sprinkling water, if recycled, should be replaced upon reaching pH = 4,5 and at the latest after one operating week (168 h).

For method A, the water sprayed on specimen surfaces shall have a silica level below 0,2  $\mu$ g/g. A combination of deionization and reverse osmosis may be used to produce water of the desired quality.

For method B, the acidic solution with a pH of 3,5 shall be in accordance with the specifications given in 4.2.

**4.7.4** Acid spraying device, either a manually operated pressure atomizer or an automated spray device, capable of spraying the acid solution, for method A. In order to produce a uniform pattern of small damage spots, the sprayer shall be adjusted to produce small droplets and the amount of acid solution applied shall be

such that the droplets do not coalesce after application. The pH of the acid solution shall be checked periodically to make sure it meets the requirements of the method used.

**4.8 Specimen holders**, either in the form of an open frame, leaving the back of the specimens exposed or providing the specimen with a solid backing. They shall be made from inert materials that do not affect the test results, for example non-oxidizing alloys of aluminium or stainless steel. Brass, steel or copper shall not be used in the vicinity of the test specimens. The backing used and a space between the backing and the test specimen can affect the results, particularly with transparent specimens, and shall be agreed on between the interested parties.

**4.9 Apparatus to assess changes in properties**, as required by the relevant standard describing determination of the properties selected for monitoring. The apparatus used for the property measurements shall be included in the test report.

NOTE ISO 4582 provides information about property measurement before and after exposure in weathering tests.

### 5 Test specimens

Test specimen should be prepared as described in ISO 4892-1, with the following additions.

The size of specimens shall be such that the condition of the specimens can be unambiguously ascertained before and after the exposure tests.

For method A, the minimum specimen size shall be 30 cm<sup>2</sup> in area, with the smallest edge at least 50 mm in length.

NOTE 1 For minimum specimen size, the amount of acid solution is about 0,1 g. This amount results from the running off at a vertical specimen orientation (see Figure A.2).

NOTE 2 ISO 294-3 provides information about the preparation of 60 mm × 60 mm specimens

The exposure of replicate specimens is highly recommended.

### 6 Test conditions

#### 6.1 Method A

#### 6.1.1 General

Method A is intended for plastics whose mechanical properties are seriously affected by acid exposure on the surface (e.g. plastic sheets and films, in terms of tensile strength and elongation at break). It is also applicable to plastics whose function or service life is mainly defined by the retention of the properties of its surface or of areas close to the surface and where the effect of acidic precipitation is mainly confined to this part of the material (e.g. building facades and building structures, light-permeable plastic roofing and bulk polymers, in terms of their decorative properties).

NOTE To check the synergistic action of acid and weathering, a second set of samples can be exposed to the same weathering exposure in parallel, but excluding acidic deposition. For pure acid exposure, ISO 175 can be used.

#### 6.1.2 Weathering cycle

The 24 h weathering cycle used in method A is characterized by continuous UV (apart from the brief interruption to spray the acid solution on the test specimen surface) and varying conditions of temperature and humidity. Table 1 describes conditions intended to simulate a high-humidity climate, such as might be experienced in subtropical or tropical locations. Table 2 describes conditions intended to simulate a more moderate, less humid climate, such as parts of North America or Central Europe. The two conditions differ in

the values of the relative humidity during the drying phase after application of the acid solution. If it is shown that the photodegradation of the specimens is not sensitive to humidity and to varying acid concentration, the test may be performed without controlling relative humidity (Table 3).

NOTE 1 As the acid concentration process is controlled by the relative humidity, this can lead to worse repeatability.

When running the test cycles described in Tables 1 to 4, programme the device to operate at the set point values for each of the controlled parameters.

Spraying the surface of the plastic specimens with the acid solution is the first step in the cycle. This spraying is performed for five days a week, followed by two daily cycles without application of the acid solution. If a uniform pattern of pinhole damage spots is aimed for, apply the acid solution to test specimens using a sprayer adjusted to provide very small droplet size and apply the solution uniformly over the specimen surface such that there is no runoff or coalescence of the droplets. Figure A.2 is a photograph showing typical appearance of the droplets after application of the acid solution.

Unless otherwise specified, use a solution with a pH of  $1.5 \pm 0.2$  for testing plastics. If a different pH is used, the pH of the applied solution shall be included in the test report.

NOTE 2 The size of the droplets of the acid solution sprayed on the surface of the test specimens affects the size of the defects produced when the droplets dry and concentrate the acid. As for larger defects, the damage concentrates in the margins, and the use of smaller droplets results in both a more concentrated damage and a more homogeneous damage pattern. At vertical exposure, due to the running off, the acid amount is limited in an expedient quantity and a visual control of the spraying pattern is enabled.

In the case of an automated spraying device for the acid, control the amount sprayed as well as a sufficiently small droplet size (see Figure A.2).

NOTE 3 If the specimens are individually sprayed outside the chamber, they can have different surface temperatures at the moment of their respective spraying, due to respective, different cooling duration.

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The surface temperature during spraying can influence the results? In this pase, the sequence of samples during spraying should be changed from one day to the next in order to average out the respective surface temperature variation and the resulting effects.

With the spraying device used, the tester should regularly check the quantity of acidic solution to be sprayed per unit of area. One possibility is weighing.

If a manual sprayer is used to apply the acid solution, spray specimens outside the test chamber. If possible, return the test specimens to the chamber within 5 min of spraying the acid solution. Do not open the test chamber enclosure, except for retrieval and placement of test specimens.

NOTE 4 Seeing that in fluorescent UV lamp devices, the difference between black standard temperature and the chamber air temperature is very small, the relative humidity at the specimen's surface is nearly the same as in the chamber atmosphere.

NOTE 5 As in fluorescent UV lamp devices, the difference between black standard temperature and the chamber air temperature is below 2 °C, temperature control can be carried out by either BST or chamber air temperature. Alternatively, black panel temperature can be used in fluorescent UV lamp devices.

NOTE 6 With the test cycle in Table 3, for devices without humidity control, there is still limited experience.