

BS ISO 29664:2010



BSI Standards Publication

# Plastics — Artificial weathering including acidic deposition

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**National foreword**

This British Standard is the UK implementation of ISO 29664:2010.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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## Plastics — Artificial weathering including acidic deposition

*Plastiques — Vieillissement artificiel y compris les dépôts acides*



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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*.

## 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  $\text{CaCO}_3$ <sup>[7][8]</sup> 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.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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  $(25 \pm 2)^\circ\text{C}$ , add 10,6 g of  $\text{H}_2\text{SO}_4$ , 3,18 g of  $\text{HNO}_3$  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.

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.

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  $45 \text{ W}\cdot\text{m}^{-2}$  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  $0,51 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$  at 340 nm or  $60 \text{ W}\cdot\text{m}^{-2}$  in the 300 nm to 400 nm bandpass. 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 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 %  $\text{H}_2\text{SO}_4$ .

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  $\mu\text{g/g}$ . 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.

The surface temperature during spraying can influence the results. In this case, 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.

**Table 1 — Test conditions for a 24 h cycle of method A, type “J”, pH 1,5**

| Step |                               | Duration | Fluorescent UV lamp apparatus  | Xenon arc lamp apparatus   |
|------|-------------------------------|----------|--|--|
| 1    | Spraying                      | < 5 min  |  |  |
| 2a   | First drying period           | 9 h      | $T = 35\text{ °C}$<br>$U = 75\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 55\text{ °C}$<br>$U = 90\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 2b   | First drying period continued | 5 h      | $T = 60\text{ °C}$<br>$U = 40\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 75\text{ °C}$<br>$U = 90\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 3    | Rain period                   | 4 h      | Continuous spraying of the specimen surface with demineralized water<br>Chamber air temperature: 35 °C   |  |
| 4    | Second drying period          | 6 h      | $T = 60\text{ °C}$<br>$U = 40\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 75\text{ °C}$<br>$U = 90\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |

“J” stands for Jacksonville (Florida) climate, for the simulation of high-humidity climates. The complete cycle (steps 1 to 4) shall be performed five days a week, followed by two daily cycles without spraying (step 1).

$T$  chamber temperature  
 BST black standard temperature, as defined in ISO 4892-1  
 $U$  relative humidity in the chamber  
 $E$  UV irradiance in the range up to 400 nm  
 $E_{\lambda}$  spectral UV irradiance at 340 nm

**Table 2 — Test conditions for a 24 h cycle of method A, type “M”, pH 1,5**

| Step |                               | Duration | Fluorescent UV lamp apparatus  | Xenon arc lamp apparatus   |
|------|-------------------------------|----------|--|--|
| 1    | Spraying                      | < 5 min  |  |  |
| 2a   | First drying period           | 9 h      | $T = 35\text{ °C}$<br>$U = 30\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 55\text{ °C}$<br>$U = 50\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 2b   | First drying period continued | 5 h      | $T = 60\text{ °C}$<br>$U < 10\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 75\text{ °C}$<br>$U < 20\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 3    | Rain period                   | 4 h      | Continuous spraying of the specimen surface with demineralized water<br>Chamber air temperature: 35 °C   |  |
| 4    | Second drying period          | 6 h      | $T = 60\text{ °C}$<br>$U < 10\text{ % RH}$<br>$E = 45\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $BST = 75\text{ °C}$<br>$U < 20\text{ % RH}$<br>$E = 60\text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |

“M” stands for medium climate, for the simulation of moderate, less humid climates. The complete cycle (steps 1 to 4) shall be performed five days a week, followed by two daily cycles without spraying (step 1).

$T$  chamber temperature  
 BST black standard temperature, as defined in ISO 4892-1  
 $U$  relative humidity in the chamber  
 $E$  UV irradiance in the range up to 400 nm  
 $E_{\lambda}$  spectral UV irradiance at 340 nm

**Table 3 — Test conditions for a 24 h cycle of method A, type “U”, pH 1,5**

| Step   |                               | Duration | Fluorescent UV lamp apparatus   |
|--|-------------------------------|----------|---|
| 1  | Spraying                      | < 5 min  |   |
| 2a   | First drying period           | 9 h      | $T = 35\text{ °C}$<br>$U$ not controlled<br>$E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 2b   | First drying period continued | 5 h      | $T = 60\text{ °C}$<br>$U$ not controlled<br>$E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| 3  | Rain period                   | 4 h      | Continuous spraying of the specimen surface with demineralized water<br>Chamber air temperature: 35 °C    |
| 4  | Second drying period          | 6 h      | $T = 60\text{ °C}$<br>$U$ not controlled<br>$E_{\lambda} = 0,76\text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| “U” stands for uncontrolled humidity. The complete cycle (steps 1 to 4) shall be performed five days a week, followed by two daily cycles without spraying (step 1). |                               |          |   |
| $T$ chamber temperature  |                               |          |   |
| BST black standard temperature, as defined in ISO 4892-1   |                               |          |   |
| $U$ relative humidity in the chamber   |                               |          |   |
| $E_{\lambda}$ spectral UV irradiance at 340 nm   |                               |          |   |

### 6.1.3 Test duration

The lifetime of products that are susceptible to degradation by acid deposition in polluted climates shows enormous variation because the acidic precipitation occurs sporadically and shows large variations in pH. Therefore, results from tests conducted according to this International Standard cannot be used to predict service life. The results from tests conducted according to this International Standard should be useful for comparing different products for relative suitability for use in highly polluted environments where acid deposition occurs.

NOTE 1 A duration of 28 d for method A, type J, pH 2,5 corresponded to a 98 d outdoor exposure in Jacksonville (Florida) concerning the damage ranking for a specific set of 20 coatings<sup>[9]</sup> (see Annex A).

Unless otherwise specified, expose specimens according to method A for four weeks. Longer exposures may be necessary, if the differences of the measured property within the set of samples are smaller than the uncertainty of its measurement.

NOTE 2 If the exposure duration is too long, differences between materials can be less than if the materials were evaluated at a shorter time. If there are only small differences between materials and all show significant damage after the recommended 4 weeks' exposure, a repeat of the test for a shorter duration can show larger differences between materials.

Interruptions to the test procedure are permissible; the storage of the specimens until resumption of the test shall take place under standard atmospheres 23/50 or 27/65, in accordance with ISO 291. If a break (longer than 20 min) occurs during the first drying phase, the acid left on the specimens shall be removed by thorough rinsing with demineralized water. In such cases, the test shall be resumed by re-spraying the specimens with the acidic solution. Each interruption shall be described in detail in the test report.

At the end of the test, no acid shall remain at the surface. In addition, because water absorption can influence the results of property measurements taken after the exposure is completed, it is important the exposure be stopped during step 4.

NOTE 3 If stabilizers are damaged by exposure to weathering with acid deposition, plastics can be more susceptible to photodegradation in subsequent weathering exposures.

## 6.2 Method B

### 6.2.1 General

Method B is intended for products made with polymers that are able to absorb or retain large amounts of moisture (e.g. textiles or geotextiles made with polymer fibres). When these products are subjected to acid rain, the acid can concentrate when the product dries, causing potential for acid-induced damage.

### 6.2.2 Weathering cycle

In method B, the acid deposition takes place in a 4 h-long period of spray with a pH 3,5 acid solution instead of a single daily application of a stronger acid solution, which is used in method A. Recycling of the acid rain is recommended. Method B shall also be carried out with continuous UV radiation. The procedure for method B is shown in Table 4.

Table 4 — Test conditions for a 24 h cycle of method B

| Step  |               | Duration | Fluorescent UV lamp apparatus  | Xenon arc lamp apparatus  |
|---|---------------|----------|--|---|
| 1   | Rain period   | 4 h      | Continuous sprinkling of the specimen surface with acid solution (pH = 3,5 ± 0,2)<br>Chamber air temperature = 35 °C<br>$E = 45 \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ | $E = 60 \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$  |
| 2   | Drying period | 20 h     | $T = 60 \text{ °C}$<br>$U < 10 \text{ % RH}$<br>$E = 45 \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,76 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$   | $\text{BST} = 75 \text{ °C}$<br>$U < 20 \text{ % RH}$<br>$E = 60 \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = 0,51 \text{ W}\cdot\text{m}^{-2}\text{nm}^{-1}$ |
| <i>T</i> chamber temperature<br><i>BST</i> black standard temperature, as defined in ISO 4892-1<br><i>U</i> relative humidity in the chamber<br><i>E</i> UV irradiance in the range up to 400 nm<br><i>E<sub>λ</sub></i> spectral UV irradiance at 340 nm |               |          |  |   |

NOTE Seeing that in fluorescent UV lamp devices, the difference between BST 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.

### 6.2.3 Test duration

Unless otherwise specified, conduct the method B exposures for six weeks. Longer exposures may be necessary if the differences of the measured property within the set of samples are smaller than the uncertainty of its measurement.

Interruptions to the test procedure are permissible; until resumption of the test, the specimens shall be stored at 3 °C to 5 °C. In the case of an interruption, do not rinse specimens with demineralized water prior to storage because the diffusion of acid into deeper layers of the specimen could be affected. Storage at the given low temperatures retards possible reactions of the acid with the specimen. If the interruptions should exceed 48 h in total, the specimens shall be discarded and the test repeated. Each interruption shall be described in detail in the test report.

NOTE Especially if stabilizers are damaged, after performing this test, some materials can have an increased sensitivity to ageing. Subsequent weathering tests can therefore lead to stronger property changes than are usual.



### 6.3 Allowed deviations in temperature and humidity

The values in Tables 1 to 4 are set point values. Correspondingly, the relative humidity may fluctuate by a value of  $\pm 5$  % RH at 30 % RH and at 40 % RH respectively  $\pm 8$  % RH at 75 % RH.

In fluorescent UV lamp devices, due to temperature inhomogeneity inside the chamber, deviations of  $\pm 2$  °C in temperature shall be allowed. Correspondingly, if controlled, the relative humidity may fluctuate by a value of  $\pm 5$  % RH at 30 % RH and at 40 % RH respectively  $\pm 8$  % RH at 75 % RH.

For xenon arc lamp devices, during equilibrium conditions, the black standard temperature may fluctuate by a value of  $\pm 2$  °C and, if controlled, the relative humidity of the chamber air by a value of  $\pm 10$  % RH.

These allowed deviations correspond to the spatial spreading inside the chamber climate.

### 6.4 Measurement of radiant exposure

If used, mount the radiation detector such that the radiometer indicates the irradiance at the exposed surface of the test specimen.

When radiant exposures are used, express the exposure interval in terms of incident radiant energy per unit area of the exposure plane, in joules per square metre ( $\text{J}\cdot\text{m}^{-2}$ ) in the measured wavelength range or joules per square metre per nanometre ( $\text{J}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ ) for the wavelength selected (e.g. 340 nm).

### 6.5 Determination of changes in properties after exposure

The changes in properties after exposure shall be determined as specified in ISO 4582.

## 7 Precision

No relevant precision data are available. It is intended to add a precision statement, once “intercomparison” data are obtained.

Precision data for coatings are given in Annex B for information only.

## 8 Test report

The test report shall contain the following information.

a) Specimen description:

- 1) a full description of the specimens and their origin;
- 2) compound details, cure time and temperature, if appropriate;
- 3) a complete description of the method used for preparation of the test specimens.

NOTE If exposure tests are conducted by a contracting agency, specimens are usually identified by code number. In such cases, it is the responsibility of the originating laboratory to provide the complete specimen description when reporting the results of the exposure test.

b) Description of exposure test, including:

- 1) a description of the method used to mount the specimens in the exposure frame, including a description of any material used as backing for the test specimens;
- 2) a description of the exposure device and radiation source;

- 3) the type of instrument used to measure the humidity, if used;
  - 4) a description of the radiometer used for measuring the radiant exposure, if used;
  - 5) if used, the type of black standard temperature sensor used and the exact position of the sensor if it was not located in the test specimen exposure area;
  - 6) the procedure for test specimen repositioning, if used;
  - 7) deviations from the test procedures.
- c) Test results:
- 1) a complete description of the test procedure used for measurement of any properties reported;
  - 2) the results, presented in accordance with ISO 4582, and including
    - the results from property measurements on the test specimens,
    - the results from property measurements on control specimens, if used,
    - the results from property measurements on unexposed film specimens, if determined, and
    - the exposure period (either the time in hours, or the radiant exposure in  $\text{J}\cdot\text{m}^{-2}$  and the bandpass in which it was measured).
- d) The start and end date of the test.

## Annex A (informative)

### Background information

#### A.1 Acidic precipitation — Outdoor conditions

The term “acidic precipitation” describes deposits on surfaces that start in the aqueous form with pH lower than 5,6. As water evaporates, the acid concentration increases, resulting in pH values that can be very low. The acid precipitation begins when air pollutants combine with water, oxygen and dust particles in the atmosphere. The primary focus is on sulfur dioxide, a by-product of the combustion of high sulfur fuels. The sulfur dioxide combines with water to form sulfuric acid and comprises 60 % to 70 % of the acidity. Other acids that have been observed are nitric acid and hydrochloric acid. From mean values of acid deposits analysed from highly polluted industrial areas, the mass ratio of the acids is generally  $\text{H}_2\text{SO}_4:\text{HNO}_3:\text{HCl} = 1,0:0,3:0,17$ <sup>[11][12][13]</sup>.

Taking into account the recorded frequency of the acidity of aqueous acidic precipitation observed outdoors, the following rough ranking of categories of acidic exposure situations can be obtained:

- dew up to pH 1,5;
- fog up to pH 2,5;
- rain up to pH 3,5.

The properties of sulfuric acid are an important reason for the damage potential of acid deposition. Sulfuric acid does not evaporate in the typical environmental conditions experienced in many locations. If the acid-containing deposit is not washed away by rain, and water evaporates, the concentration of the sulfuric acid can increase to between 50 % and 60 %, controlled by the relative humidity. When deposits develop such high acid concentration in the presence of solar radiation, the potential for producing damage to the polymer surface is increased. Because of their similar mechanisms, the acid dew and fog (ADF) test can also be used without modification for simulating the effect of dusts and aerosols containing acids.

Outdoor weathering tests<sup>[14]</sup> and artificial weathering tests demonstrated a synergy between acidic precipitation and the solar UV radiation<sup>[15][16]</sup>. On the other hand, at least temporary antagonistic effects are possible, as photocrosslinking of the polymer matrix can increase the acid resistance of the material<sup>[17]</sup>.

#### A.2 Experiences with methods A and B

Methods A and B in literature are introduced as the acid dew and fog test (ADF test) and the acid rain test (AR test), respectively.

Extensive experimental tests showed that temperature and humidity can have a considerable effect on the ranking of damage. For the ADF test the temperature and humidity values were optimized to fit the averaged ranking of three summer seasons of the outdoor exposure in Jacksonville, Florida (variant J), respectively six years in Central Europe (variant M).

The ADF test variant, ADF J, with pH 2,5 was assessed on the basis of 16 modern automotive finishes (base-coat/clearcoat systems with black basecoat) of different types and from different manufacturers in comparison with a conventional 14-week outdoor weathering test in Jacksonville. The result achieved after 28 days with the ADF test, in terms of the ranking, correlates with the mean result from three outdoor weathering tests conducted at the same time of year (end of May to the beginning of September) in 1999 to 2001 with a ranking correlation coefficient of 0,9. The reference criterion was the increase in haze.

Testing conducted according to method A was used to evaluate nine different polymer systems intended for use as automotive antenna bases. The results from the method A test were compared to results from a one-year period of outdoor exposure in Berlin, Germany. The rank correlation between results from the method A exposures and those from the outdoor exposure in Berlin was 0,85. The rank correlation coefficient for the same materials between a conventional artificial accelerated test conducted in accordance with ISO 4892-3 and those from the Berlin exposure was 0,48. The criterion was the visual assessment done by 10 persons<sup>[17]</sup>.

The AR test was assessed on a series of samples of different geotextiles based on polyolefins, PET, and PA. They were subjected to six-month outdoor weathering in Berlin (Germany) and Bandol (Southern France) and, for comparison, to artificial weathering methods without acid exposure (conforming to ISO 4892-1, ISO 4892-2 and ISO 4892-3) and with acid exposure (ADF test, AR test). Subsequently, the weathering-related decrease in the residual tensile strength was determined. As a whole, there was a satisfying correlation between the weathering results exposed outdoors. Only PA showed a stronger decrease of the retained strength in Berlin than in Bandol. REM images of the PA fibres showed surface cracks after the Berlin exposure, which were caused by pollutants or acidic precipitation during the outdoor exposure. Of the artificial weathering methods, only the AR test yielded the same strength ratings as those obtained with outdoor weathering. The results of the artificial weathering processes without acid exposure and the ADF test (ADF M with pH 2,5) did not correlate with outdoor weathering<sup>[17]</sup>.

### A.3 Orientation of the specimens

Due to the construction of weathering devices, both horizontal and vertical specimen mounting are allowed.

However, vertical specimen orientation has the advantage that generally (due to the running off) the acid amount is limited in an expedient quantity. It prevents acid droplets from combining into wetted areas that are too extended. This would produce damaged areas of too large a size and widen the size distribution to too great an extent. Consequently, vertical specimen orientation leads to a homogeneous and ventilation-independent damage pattern, which is especially important for specimens used for mechanical tests (testing, e.g. dumbbell-shaped specimens). Thus, the reproducibility is better than for horizontal exposure.

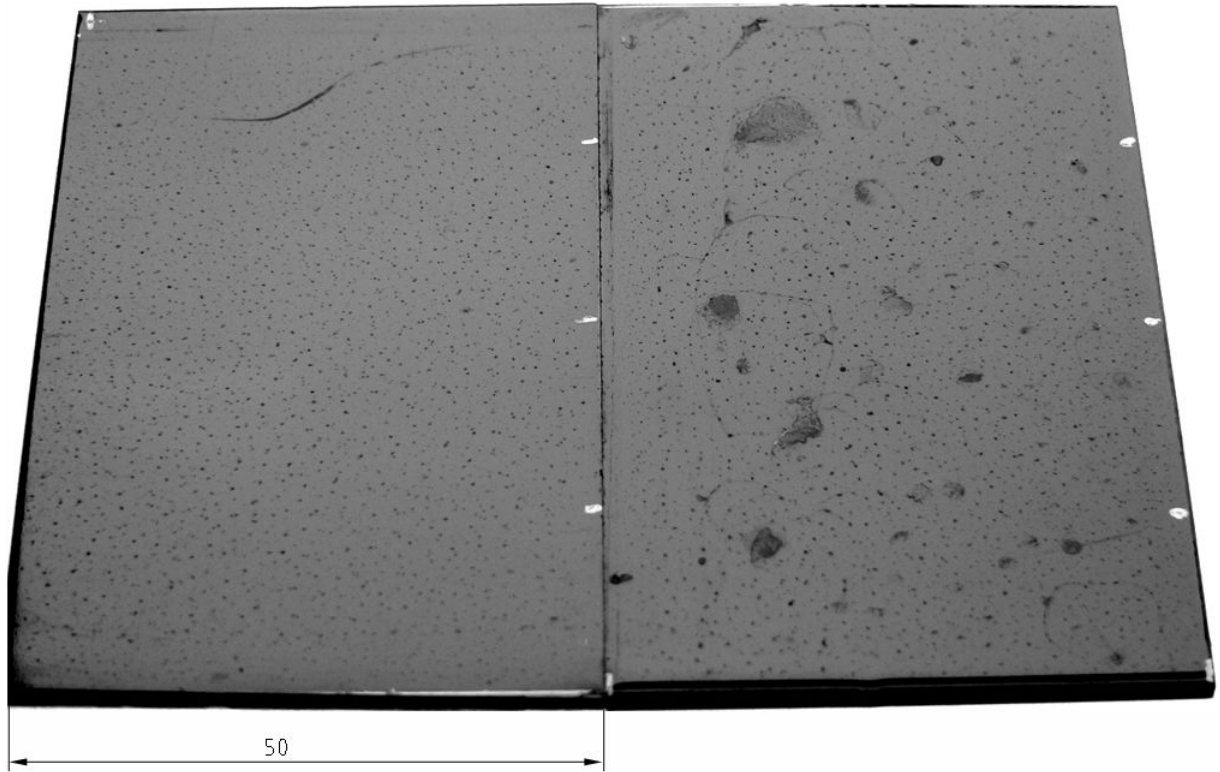
For a fluorescent device conforming to ISO 4892-3, a comparison was made to a similar device using 45° reflecting mirrors which directed the radiation to horizontally mounted specimens. For one specimen, the typical homogeneity of the damage for vertically and horizontally mounting is shown in Figure A.1. The small pinhole-like acid-damaged areas were found for both exposures, while the larger acid damage, formed after coagulation of droplets into larger areas, only occurred for the horizontal exposure.

Although the damage patterns for horizontally mounted specimens look more like the damage experienced in outdoor exposure in terms of their size, upon evaluating the ranking for the whole sample set, no significant improvement in the ranking correlation to outdoor weathering was found. This suggests that there is no difference in the damage mechanism, which is confirmed by electron beam microanalysis, which maps sulfur in both the smaller-sized damaged areas as well as the larger-sized areas, which only occur in the horizontal mounting.

Showing a better homogeneity and, therefore, smaller statistic deviations, a vertical specimen orientation allows smaller specimen sizes, which makes the throughput of more specimens possible. Additionally, homogeneous damage patterns are essential, if mechanical properties are considered.

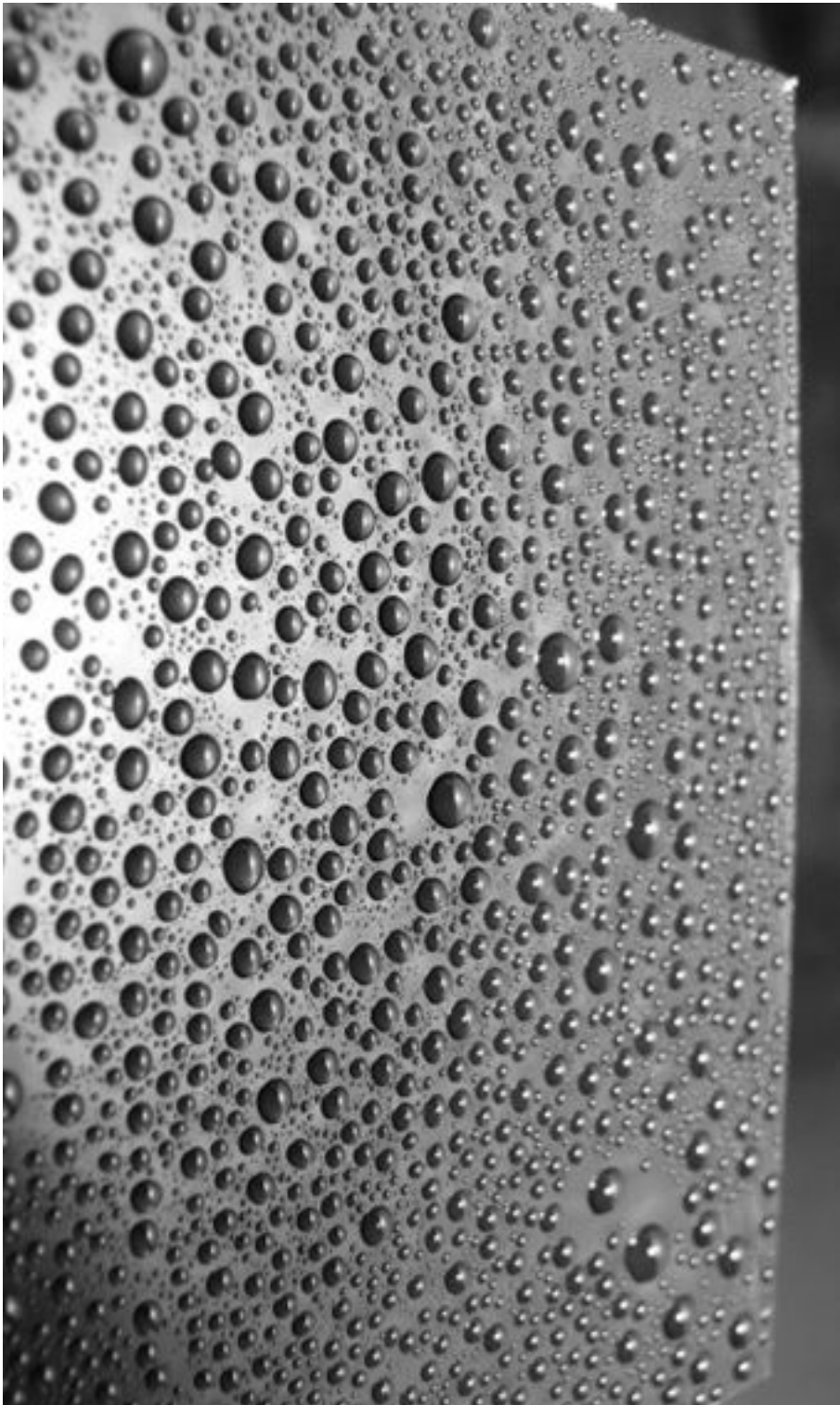
There is wide experience in the use of vertical specimen orientation available<sup>[9][10][11][15][16][17]</sup>.

Dimensions in millimetres



**Figure A.1 — Comparison of damage homogeneity for vertically (left) and horizontally (right) mounting**

Figure A.2 shows a typical droplet pattern after acid spraying in the ADF test, with vertical specimen orientation.



**Figure A.2 — Typical droplet pattern after acid spraying (70 mm specimen height)**

## Annex B (informative)

### Precision data for coatings

#### B.1 General

Repeatability data are available only for coatings for the ADF test with pH 2,5 in fluorescent UV lamp devices.

Data about the reproducibility are not yet available.

#### B.2 Repeatability

Major sources of variation in the results of the ADF test are:

- fluctuations in the composition and acidity of the artificial acidic precipitation (weighing, volume measurement and pH measurement during its preparation);
- fluctuations in the constancy of the quantity of sprayed acid (wear of the spraying device, subjectively caused fluctuations during the manual execution of the spraying process);
- fluctuations in adherence to the climatic conditions (particularly temperature, humidity, and duration of the joint action of acid and UV radiation).

The repeatability was determined using a fluorescence UV lamp apparatus in five tests with four selected automotive finishes, repeated at intervals of up to three years. In this period, new batches of the artificial acidic precipitation were mixed several times and the person performing the spraying process and the weathering device was changed. The ascertained variations in loss of gloss and increase in haze were of a statistical nature and were within the range of results of the individual measurements distributed in each case over the specimen's surface. Absolute variations in loss of gloss of four gloss units (with a standard deviation of  $\sigma = \pm 1,4 \%$ ) and an increase in haze of  $\pm 10$  haze units (with a standard deviation of  $\sigma = \pm 8$  haze units) can be expected.

Repeatability data for the ADF test using xenon arc apparatus are not available.

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