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**Paints and varnishes — Artificial
weathering including acidic deposition**

Peintures et vernis — Vieillessement artificiel comportant un dépôt acide



Reference number
ISO 15110:2013(E)

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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 15110 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

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Introduction

This International Standard specifies a method of simulating the damaging effect of outdoor weathering with relevance to acidic atmospheric precipitation on polymeric products. In addition to the classical environmental variables of temperature and humidity, acidic atmospheric precipitation can also have a significant effect on the photochemical ageing of polymers, e.g. by attacking the stabilizers. 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.

The method is based on VDI Guideline VDI 3958-12.[\[9\]](#)

Paints and varnishes — Artificial weathering including acidic deposition

1 Scope

This International Standard specifies a so-called acid dew and fog test (ADF test) as an accelerated laboratory test method for simulating, by the use of artificial acidic precipitation, the damaging effects of acidic atmospheric precipitation in association with UV radiation, neutral condensed precipitation, and changing temperature and humidity. This test method is intended to be used in evaluating, on the basis of relative performance rankings, the suitability of polymeric materials for use in outdoor environments with acidic precipitation. It is not intended to generate the same extent of damage or the same damage pattern as in outdoor weathering, but rather to give a ranking which is similar to that which would be obtained in outdoor weathering. The method produces damage which is more homogeneous, allows fewer specimens to be exposed (and hence more rapid testing) and enables evaluation of the exposed specimens to be carried out using methods which are more objective than visual assessment.

2 Normative references

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

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

ISO 4892-2:2006, *Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

ISO 4892-3:2006, *Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

3 Principle

The combined action of solar UV radiation, heat, humidity, wetting and acidic deposition is simulated in weathering devices similar to those described in ISO 4892-2 and ISO 16474-2 for xenon-arc lamps, and in ISO 4892-3 and ISO 16474-3 for fluorescent UV lamps.

Included in the artificial exposure is an acidic exposure, which is accomplished by a once-per-day spraying of acid. The aim of this test is not to reproduce the visual damage patterns which result from outdoor exposures, which show a very widespread distribution that is hard to evaluate. Rather, the aim is to reproduce the same ranking which would be obtained with an outdoor exposure, by causing damage by the same mechanisms but creating a homogeneous distribution on a smaller scale that can be evaluated in an objective way.

Accelerated weathering is achieved by the serial arrangement of the worst conceivable combinations of environmental conditions for the object under test (referred to hereafter as the “worst-case scenario”). This is accomplished by reproducing these environmental conditions in a more compressed sequence than would be the case outdoors and by including artificial acidic precipitation. However, the values of the environmental conditions used do not significantly exceed those encountered in practice outdoors.

4 Apparatus

4.1 General

For this test, weathering devices as specified in either ISO 4892-2 or ISO 4892-3 shall be used. The test chamber, as well as the specimen holders, shall be made of acid- and UV-resistant materials.

4.2 Laboratory radiation sources

4.2.1 Fluorescent UV lamp apparatus

Fluorescent UV lamp apparatus shall be in accordance with that specified for ISO 4892-3:2006, method A, using type 1A lamps. For a fluorescent UV lamp apparatus giving the spectral distribution specified for ISO 4892-3:2006, method A, a UV irradiance of $E_{UV} = (45 \pm 5) \text{ W/m}^2$, constant over time and uniformly applied to the specimens in the weathering device, shall be achieved in the spectral range 290 nm to 400 nm.

4.2.2 Xenon arc lamp apparatus

Xenon-arc lamp apparatus shall be in accordance with that specified for ISO 4892-2:2006, method A. Irradiance uniformity shall be in accordance with the requirements specified in ISO 4892-1. For a xenon-arc lamp apparatus, equipped with daylight filters, giving the spectral distribution specified for ISO 4892-2:2006, method A, the UV irradiance shall be $E_{UV} = (60 \pm 5) \text{ W/m}^2$ in the spectral range 300 nm to 400 nm.

4.3 Test chamber

The design of the test chamber may vary, but it shall be constructed from inert material, meeting the requirements of ISO 4892-1 and ISO 4892-2 or ISO 4892-3, respectively. The test chamber shall provide for control of temperature or black standard temperature and for humidity control that meets the requirements of ISO 4892-1. Devices shall also be provided to produce water spray. Water used for the water spray shall meet the requirements of ISO 4892-1. The weathering device shall be capable of completing the change from each climatic phase to the next during the ADF test within 30 min.

4.4 Wetting system

The test chamber shall be equipped with a means of introducing an intermittent spray of water onto the front of the test specimens, under specified conditions. The spray system shall be made from corrosion-resistant materials that do not contaminate the water employed.

The water spray system in xenon-arc lamp instruments shall be as specified in ISO 4892-2.

4.5 Specimen holders

The specimen holders shall be made from inert materials that will not affect the test results, for example non-oxidizing alloys of aluminium, or stainless steel. Brass, steel or copper shall not be used.

4.6 Black-standard thermometer

The black-standard thermometer used shall comply with the requirements for these devices that are given in ISO 4892-1.

4.7 Humidity sensor

Use a hygrometer for measuring the relative humidity.

4.8 Acid spray device

A manually operated atomizer or an automatic spraying device may be used.

4.9 Radiometer

When a radiometer is used, it shall comply with the requirements of ISO 4892-1.

5 Solutions for artificial acidic deposition

Prepare an acid mixture with a pH of 1,5 at (25 ± 2) °C by dissolving 10,6 g of H₂SO₄, 3,18 g of HNO₃ and 1,80 g of HCl in 10 l of demineralized water (this gives a mass ratio of 1,0:0,3:0,17). These quantities refer to acids with a calculated concentration of 100 %. Depending on the mass concentrations of the acids available, larger quantities of acid might have to be weighed out (e.g. if 36 % HCl is used, the quantity will have to be $1,8/0,36 = 5,0$ g). Analytical-grade acids shall be used.

Then prepare a solution with a pH of 2,5 by diluting the pH 1,5 solution with demineralized water by a volume ratio of 1:10.

The pH 2,5 solution is the recommended solution. However, the more aggressive pH 1,5 solution may be used if agreed upon by the interested parties. If the pH 1,5 solution is used, this shall be mentioned in the test report.

Before starting the exposure, check the pH-value of the solution using, for instance, pH test strips. If it deviates by more than pH 0,3 from the theoretical value, replace the solution.

To avoid changes in the pH-value of the solution, store it in an airtight container.

Acid spraying may be carried out either manually or automatically (see 8.3).

6 Test specimens

6.1 General

All specimens shall be permanently marked.

NOTE The specimens will be delivered by a customer or specifically prepared for this test or taken from a building component. Guidelines for preparation are given in ISO 1514. Guidelines for sampling are given in ISO 15528.

6.2 Specimen size

The specimen size shall be such that the condition of the specimens can be unambiguously ascertained before and after the ADF test.

For vertically exposed specimens, due to the homogeneity of the damage, only relatively small areas need to be evaluated. The minimum exposed surface area shall be 30 cm², with the shortest edge at least 50 mm in length to reduce local variation in the acid deposition due e.g. to the spraying procedure or edge effects.

6.3 Corrosion of the substrates

If possible, the substrate used should be the same as that to which the coating material will be applied in practice. It shall be made, as far as possible (see below), from inert material.

If the possibility of corrosion or delamination at the edges cannot be excluded, suitable edge protection should preferably be provided. Also, exposed edges and substrate surfaces that might react with acidic precipitation (e.g. those made of non-stainless steels, aluminium, copper, zinc or concrete) should be suitably protected.

6.4 Reference specimens

The use of reference specimens is recommended for assessing the repeatability. Reference specimens of the same type of material as employed in previous test series should be used.

If possible, these reference specimens should be made of a materials which do not change with time (i.e. do not age) under normal storage conditions.

7 Designation of type of test

Exposures carried out under conditions corresponding to the subtropical climate of Jacksonville in Florida (i.e. those of type J as defined in [Table 1](#)) are designated:

ISO 15110, ADF-J test

Exposures carried out under conditions corresponding to a moderate climate like that of central Europe (i.e. those of type M as defined in [Table 2](#)) are designated:

ISO 15110, ADF-M test

8 Procedure

8.1 General

The ADF test may be performed in weathering devices as described in ISO 4892-1, ISO 4892-2 or ISO 4892-3.

If damage occurs unexpectedly fast, it is recommended that a check be carried out to determine if the damage really results from the synergistic action of the acid and the weathering, by performing separate acid and weathering exposures. For the acid-free weathering exposure, the same weathering conditions should preferably be used. For the pure acid exposure, ISO 2812-5 or ISO 175 could be used.

NOTE 1 Testing in different types of weathering device can lead to different results.

NOTE 2 To differentiate between the special action of the acidic deposition and that of usual weathering effects, it can be useful to expose a second set of specimens to the same weathering exposure in parallel, but without acidic deposition.

The specimens may be mounted either in an open frame or on a solid backing.

NOTE 3 If backing is used, the presence of a space between the backing and the test specimens might affect the results, particularly with transparent specimens.

During the dry periods, specimens shall be exposed to varying relative humidities as specified in [Table 1](#) or [Table 2](#). During the rain period, specimens shall be sprayed with water in the way specified in [Table 1](#) or [Table 2](#).

8.2 Wetting and relative humidity

The water spray system in fluorescent UV lamp devices shall permit a constant rain flux density of 2 l to 5 l of water per square metre of specimen surface per second to be produced continuously with time.

The spray shall be uniformly distributed over the specimens.

If the water is recycled, the quantity of circulating water used for the ADF test shall not be less than 50 l. Because of the uptake of the acids washed off the specimens and possible contamination from the specimen material, the spray water, if recycled, shall be replaced when it reaches pH 4,5 and at the latest after 1 operating week (168 h).

The water sprayed onto the specimen surfaces shall have a conductivity below 5 $\mu\text{S}/\text{cm}$, contain less than 1 $\mu\text{g}/\text{g}$ dissolved solids and leave no observable stains or deposits on the specimens. Care shall be taken to keep silica levels below 0,2 $\mu\text{g}/\text{g}$. A combination of deionization and reverse osmosis may be used to produce water of the desired quality.

Tests carried out in accordance with this International Standard require the relative humidity to be controlled during the dry periods. The location of the sensor used to measure the humidity shall be in accordance with ISO 4892-1.

NOTE The air humidity will have a significant influence on the photodegradation of many polymers and on the effect of the acidic precipitation, as it influences evaporation and hence the concentration of the acid on the specimens.

8.3 Acid spraying

An atomizer similar to that used with perfume bottles may be used to apply acid spray to the specimens manually. This allows the amount of acid to be controlled by visually checking the spray pattern.

If an automatic spraying device is used, the device shall control the amount sprayed on to the specimens as well as the droplet size, which shall be sufficiently small.

8.4 Weathering cycles

The 24 h weathering cycle recommended for the ADF test (the ADF cycle) exposes the specimens to continuous UV radiation and changing climatic conditions. This cycle is shown in [Table 1](#) for a Jacksonville/Florida climate and in [Table 2](#) for a moderate climate with a lower relative humidity.

The first step in the ADF cycle, the spraying of the specimen surface with the artificial acidic precipitation, is performed on five consecutive days of each week, followed by two 1-day cycles without acid spraying.

To protect the chamber materials, the specimens should preferably be sprayed outside the test chamber.

After spraying, the specimens shall be returned to the test chamber. The time required for spraying and the necessary handling of the specimens shall not exceed 5 min. The test chamber of the weathering device shall remain set to the climatic test conditions and shall only be opened for the maximum time necessary for specimen removal and replacement.

The quantity of acidic solution applied shall be such that the surface becomes uniformly wet, but not so much that the solution runs off the specimen surface, even when oriented in its recommended vertical position.

It is recommended that, for a particular spraying device, the tester should regularly check the quantity of acidic solution sprayed on to unit area of the specimen surface (e.g. by measurement of the mass increase).

The various steps in the ADF cycle are shown in [Table 1](#) and [Table 2](#), respectively.

The following symbols are used:

BST	Black-standard temperature
RH	relative humidity in the chamber
E_{UV}	UV irradiance in the range up to 400 nm
E_{λ}	spectral irradiance

The complete cycle (steps 1 to 4) shall be performed five days a week, followed by two 1-day cycles without spraying (i.e. without step 1).

As, in fluorescent UV lamp devices, the difference between black standard temperature and the chamber air temperature is less than 2 °C, the temperature may be controlled either using BST or the chamber air temperature.

Table 1 — Test conditions used for the daily cycle in the type J ADF test
(where “J” stands for the subtropical climate of Jacksonville in Florida)

Step	Duration	Fluorescent UV lamp apparatus	Xenon-arc lamp apparatus	
1	Acid spraying	< 5 min	30 g to 40 g of acid solution per m ² of specimen surface preferably outside the test chamber	
2a	First dry period	9 h	BST = (35 ± 2) °C, RH = (75 ± 8) %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (55 ± 2) °C, RH = (90 ± 10) %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$
2b	First dry period continued	5 h	BST = (60 ± 2) °C, RH = (40 ± 5) %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (75 ± 2) °C, RH = (90 ± 10) %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$
3	Rain period	4 h	Continuous spraying of the specimen surface with demineralized water, Chamber air temperature: (35 ± 3) °C	
4	Second dry period	6 h	BST = (60 ± 2) °C, RH = (40 ± 5) %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (75 ± 2) °C, RH = (90 ± 10) %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$

Table 2 — Test conditions used for the daily cycle in the type M ADF test
(where “M” stands for a moderate climate like that of central Europe)

Step	Duration	Fluorescent UV lamp apparatus	Xenon-arc lamp apparatus	
1	Acid spraying	< 5 min	30 g to 40 g of acid solution per m ² of specimen surface, preferably sprayed on outside the test chamber	
2a	First dry period	9 h	BST = (35 ± 2) °C, RH = (30 ± 5) %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (55 ± 2) °C, RH = (50 ± 10) %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$
2b	First dry period continued	5 h	BST = (60 ± 2) °C, RH < 10 %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (75 ± 2) °C, RH < 20 %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$
3	Rain period	4 h	Continuous spraying of the specimen surface with demineralized water. Chamber air temperature: (35 ± 3) °C	
4	Second dry period	6 h	BST = (60 ± 2) °C, RH < 10 %, $E_{UV} = (45 \pm 5) \text{ W}\cdot\text{m}^{-2}$ or $E_{\lambda} = (0,76 \pm 0,08) \text{ W}\cdot\text{m}^{-2}\cdot\text{nm}^{-1}$ at 340 nm	BST = (75 ± 2) °C, RH < 20 %, $E_{UV} = (60 \pm 5) \text{ W}\cdot\text{m}^{-2}$

8.5 Test duration

For ADF tests of both types, a test duration of 4 weeks is recommended.

NOTE This corresponds to 20 cycles with acid spray.

Longer exposures might be necessary if the differences in a measured property between the specimens in a set of specimens are smaller than the uncertainty of measurement of the property.

The exposure shall be terminated after the second dry period (step 4), as the measurement of properties might depend on the water content (e.g. gloss measurement). Particular care shall be taken that no acid remains on the specimen surfaces.

Interruptions to the test procedure are permissible, in which case the specimens shall be stored under standard climatic conditions until resumption of the test. If a break of longer than 20 min occurs during the first dry 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 documented in detail.

8.6 Further weathering of specimens

Especially if stabilizers based on sterically hindered amines are involved in reactions with the acid during the exposure, some plastics might develop an increased sensitivity to ageing. Therefore, subsequent exposures can lead to more marked property changes than usual.

Care shall be taken if ADF-weathered specimens have to be stored for a long period of time, as any acid remaining on the specimens can continue to cause damage during storage.

9 Assessment of results

Since acidic atmospheric precipitation occurs sporadically and randomly in terms of frequency and acidity, the lifetime of identical products in outdoor use in such an environment can vary enormously. A rough estimate of a product's service life on the basis of the results of method A of ISO 4892-2:2006 or ISO 4892-3:2006 and the usually only very imprecise information on the degree of aggressiveness from acidic atmospheric precipitation in the envisaged area of use is only possible if suitable repeated outdoor weathering tests have been carried out in sufficient numbers on the same object.

NOTE 1 A duration of 28 d was chosen for the ADF-J test, using a solution of pH 2,5, in order to simulate the damage caused by a 98 d outdoor exposure in Jacksonville for a specific set of 20 coatings (see also [Annex A](#)).

However, method A of ISO 4892-2:2006 and ISO 4892-3:2006 is useful for comparing different products in terms of their suitability for use in an acid-charged environment. Therefore, it makes little sense to define the test duration as the degree of aggressiveness.

It shall be agreed between the interested parties which properties of the coating are to be measured before, during and after the exposure, and which standards are to be used for this purpose.

NOTE 2 Suitable methods are given in e.g. ISO 2813, ISO 3668, ISO 4628-1 to ISO 4628-8 and ISO 4628-10, ISO 11664-1 to ISO 11664-5 and ISO 13803.

10 Precision

10.1 General

Repeatability data are available only for the ADF test in fluorescent UV lamp devices.

Data on the reproducibility are not available.

10.2 Repeatability

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

- a) fluctuations in the composition and acidity of the artificial acidic deposition solution (weighing, volume measurement, pH measurement during its preparation);
- b) fluctuations in the quantity of acid sprayed on to the specimen surface (due to wear of the spraying device and, in the case of a manual spraying device, differences in the execution of the spraying process);

- c) non-adherence to the climatic conditions (particularly temperature, humidity, and the duration of the joint action of acid and UV radiation).

The repeatability was determined using a fluorescent UV lamp apparatus, the Weiss UV200 Global UV test unit¹⁾, in five tests on four selected automotive finishes, repeated at intervals of up to three years. During this period, new batches of the artificial acidic deposition solution were prepared several times and the person performing the spraying process and operating the weathering device changed. The variations ascertained in loss of gloss and increase in haze were of a statistical nature and were within the range of the results of the individual measurements distributed in each case over the specimen surface. Absolute variations in loss of gloss of 4 gloss units (with a standard deviation, σ , of $\pm 1,4$ %) and an increase in haze of ± 10 haze units (with a standard deviation, σ , of ± 8 haze units) can be expected.

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

11 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard (ISO 15110);
- b) the type of exposure conditions used (type J or M);
- c) all information necessary for identification of the coating material tested;
- d) a description of the test apparatus and the light source, including:
 - 1) the type of test apparatus and light source,
 - 2) a description of the filters used,
 - 3) the irradiance at the surface of the specimens (including that of the band pass filter with which the radiation was measured), as well as a description of the radiation-measuring instrument used,
 - 4) the duration of use of the filters and light source, in hours, before the beginning of the exposure;
- e) the type of instrument used to measure the air humidity;
- f) a description of the procedure used to mount the specimens on the exposure frame, including a description of all materials used to support the specimens;
- g) in the case of transparent materials, the type of backing used for the specimens;
- h) the type (manual or automatic) of spraying device used and its location (inside or outside the test chamber);
- i) the procedure used to change of the positions of the specimens in the test chamber, if applicable;
- j) the result of the assessment of the specimens, including:
 - 1) the method used to measure each of the properties determined,
 - 2) the results of the determinations on the test specimens,
 - 3) the results of determinations on any control specimens,
 - 4) the results of determinations on any unexposed reference specimens,

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this apparatus.

- 5) the UV radiant exposure, in J/m^2 , and wavelength range in which the above measurements were made;
- k) details of any deviations from the specified exposure conditions;
- l) details of any unusual features (anomalies) observed during the test;
- m) the dates of the beginning and end of the test.

Annex A (informative)

Background information

A.1 Acidic precipitation — Outdoor conditions

The term “acidic precipitation” describes the deposition of pollution on surfaces that, in its aqueous form (rain, dew, fog, snow, frost or hail) exhibits an acidity more severe than pH 5,6 and that, in the form of aerosols or dusts, contains free acids.^[1] Acidic precipitation forms when certain air pollutants combine with oxygen, water or dust particles in the atmosphere. The main focus is on sulfur dioxide, a by-product of the combustion of fossil fuel, which forms sulfuric acid (H₂SO₄) after oxidation and contributes to about 60 % to 70 % of the acidity of precipitation. Other frequently encountered components of acidic precipitation are nitric acid (HNO₃) and hydrochloric acid (HCl). From mean values derived from the mixing ratios of the above-mentioned acids recorded in major industrial regions and frequently used internationally for test purposes^[2] to^[4], the following mass ratio is derived:

$$\text{H}_2\text{SO}_4:\text{HNO}_3:\text{HCl} = 1:0,3:0,17$$

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 is obtained:

- Dew: up to pH 1,5
- Fog: up to pH 2,5
- Rain: up to pH 3,5

In outdoor exposures as well as in the ADF test, the special properties of sulfuric acid are crucial for the so-called worst-case scenario. Sulfuric acid does not evaporate in the climatic environmental conditions to be expected outdoors. Due to water evaporation, acidic precipitation deposited on the surface of an object in rain-free environmental conditions might become concentrated into sulfuric acid with a mass fraction of 50 % to 60 %, particularly if exposed to bright sunlight. Because of their generally higher acidity, the most serious surface damage is expected from exposure to acid dew or fog precipitation. The ADF test therefore chiefly simulates the effect of fog and dew precipitation in joint action with the relevant climatic conditions. Because of the similar mechanisms involved, the ADF test may also be used without modification to simulate the effect of dusts and aerosols containing acids. The worst-case outdoor weather scenario has proven to be the combination of deposited dew or fog droplets with sunlight.

Outdoor weathering tests^[5] and artificial weathering tests have demonstrated that a synergy exists between acidic precipitation and solar UV radiation.^[6]^[7] During acid exposure and thereafter, a significant increase in the destructive effect of UV radiation can be expected on at least some polymers as a result of accelerated photodegradation. On the other hand, the same UV radiation, if applied in a sufficient quantity before an acid attack, is capable — depending on the type of polymer — of either significantly weakening (e.g. with polycarbonate or PMMA) or strengthening (e.g. with automotive paint finishes) the acid resistance of the material's surface. These effects can change the ranking of the durability of different types of polymer compared with the exposure according to ISO 4892-2 and ISO 4892-3, where wetting is usually produced by spraying the test specimens with demineralized/deionized water.

A.2 Experience with the ADF test

Extensive experimental tests have shown that temperature and humidity can have a considerable effect on the ranking with respect to damage. For the ADF test, the temperature and humidity values have

been optimized to fit the average ranking for three summer seasons of outdoor exposure in Jacksonville (for the type J test) and for six years in central Europe (for the type M test).

The ADF-J test using a solution of pH 2,5 was assessed by testing 16 modern automotive finishes (base-coat/clearcoat systems with a black basecoat) of different types and from different manufacturers in comparison with a conventional 14-week outdoor weathering test in Jacksonville, Florida. 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 Pearson ranking correlation coefficient of 0,9. The reference criterion was the increase in haze.

The ADF-M test using a solution of pH 2,5 was assessed by testing 18 white aircraft finishes of different formulations in comparison with a six-year outdoor weathering test (carried out in 1995 to 2001) in an industrial area within the city of Leipzig. The mean haze obtained from the specimens subjected to outdoor weathering was achieved with the ADF test after 130 days with a Pearson correlation coefficient of 0,71.[8]

Investigations showed an only negligible improvement in the ranking correlation with outdoor weathering if the most abundant ions found in Jacksonville precipitations[4] were incorporated, in addition to the acid mixture, in the ADF test on the automotive coatings.

A.3 Orientation of the specimens

Vertical orientation of specimens in the test chamber was chosen as it is more easily achieved in most test chambers. At the same time, it avoids acid droplets combining to give too extensive wetted areas that would produce damaged areas of too large size and widen the size distribution too much.

A comparison was made between a fluorescent device as specified in ISO 4892-3 and a similar device which used 45° reflecting mirrors that directed the radiation to horizontally mounted specimens, with the following results.

The typical homogeneity of the damage for vertical and horizontal mounting is shown for one specimen in [Figure A.1](#). The small pinhole-like acid-damaged areas were found for both exposures, but the larger areas of 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, assessing the ranking for the whole set of specimen did not indicate any significant improvement in the ranking correlation with outdoor weathering. This suggests that there is no difference in the damage mechanism, which is confirmed by electron beam microanalysis that maps sulfur in the smaller-sized damaged areas as well as in the larger-sized areas that only occur with horizontal mounting.

Since it shows better homogeneity, and therefore smaller statistical deviations, vertical specimen orientation allows smaller specimens to be used.

A typical droplet pattern obtained in the ADF test after acid spraying is shown in [Figure A.2](#).

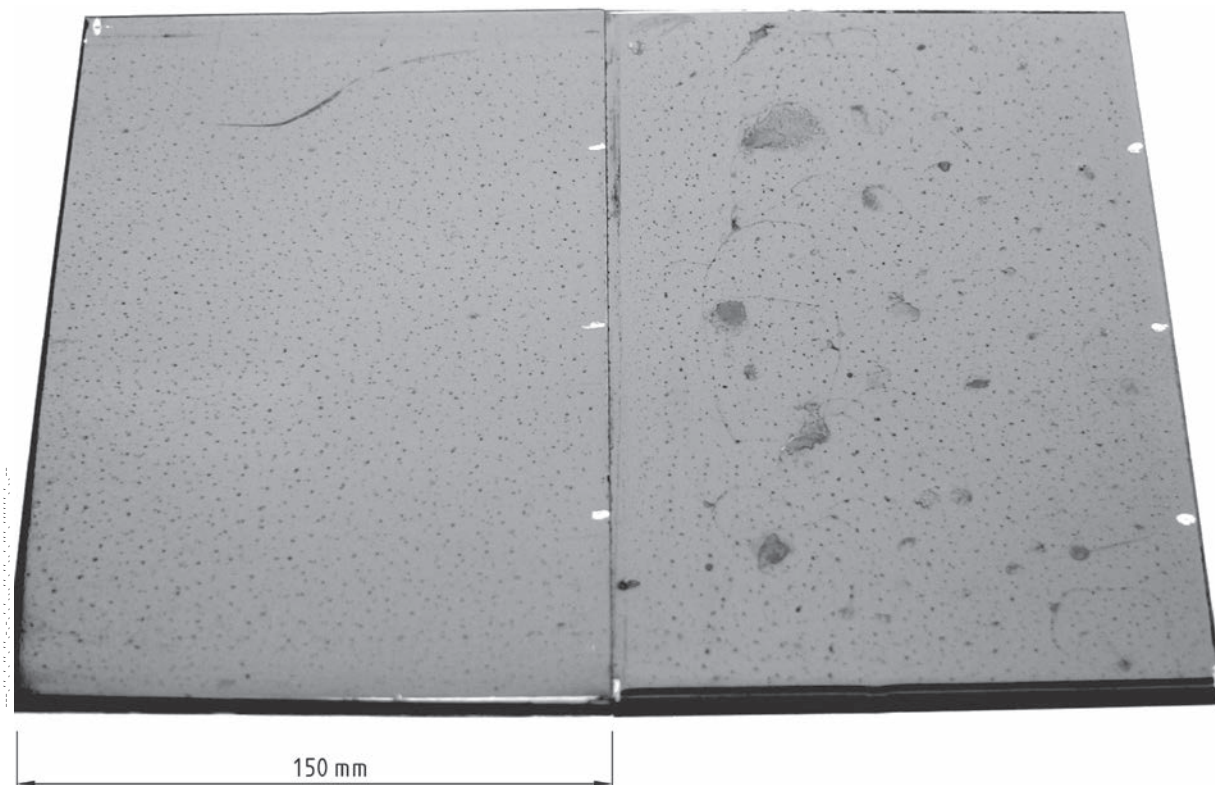


Figure A.1 — Comparison of damage homogeneity for vertically (left) and horizontally (right) mounted specimens

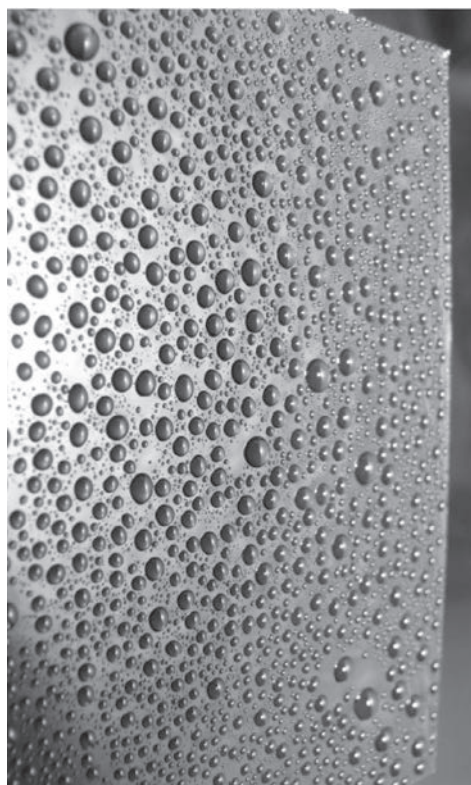


Figure A.2 — Typical droplet pattern after acid spraying (specimen height 70 mm)

Bibliography

- [1] RODGERS W.R., GARNER G.D., CHEEVER G.D. Study of the Attack of Acidic Solutions on Melamine/Acrylic Basecoat/ Clearcoat Paint Systems. *Journal of Coatings Technology* **877** (1998) 70, pp. 83-95
- [2] WOLFF C.T., RODGERS W.R., COLLINS D.C., VERMA M.H., WONG C.A. Spotting of Automotive Finishes from the Interactions Between Dry Deposition of Crystal Material and Wet Deposition of Sulfate. *Journal Air Waste Manage. Assoc.* 1990, **40** 12, pp. 1638-1648
- [3] MÖLLER D. Acid rain — gone? In: *Atmospheric Environmental Research — Critical Decisions between Technological Progress and Preservation of Nature*. Springer Verlag, Berlin, 1999, pp. 141-78.
- [4] SCHULZ U., SCHULZE R.D., SICKFELD J., TRUBIROHA P. Der Einfluss der Bestandteile saurer Niederschläge auf die photochemische Alterung von Autolacken (The influence of the constituents of acidic precipitation on the photochemical ageing of automotive paints). In: *Kongressband XXII. FATIPEC CONGRESS*, Budapest, **Vol. I**, May 1994, pp. 218-23.
- [5] LAMPE K., & SAARNAK A. Acid Rain Test. *Farbe + Lack* 1986, **92** 8, pp. 692-696
- [6] SCHULZ U., TRUBIROHA P., SCHERNAU U., BAUMGART H. The effects of acid rain on the appearance of automotive paint systems studied outdoors and in a new artificial weathering test. *Prog. Org. Coat.* 2000, **40** pp. 151-165
- [7] Schulz U., Trubiroha P., Boettger P., Bolte H. Service life prediction of polymeric building materials using the Acid Dew and Fog test. Proceedings of the 8th dbmc, Vancouver, Canada, May 1999, pp. 864-872
- [8] Wachtendorf V., Schulz U., Trubiroha P. Adaption of the Acid Dew and Fog (ADF) Test for Service Life Prediction of Aircraft Coatings. 1st European Weathering Symposium, Prague, Sept. 2003. In: *Natural and Artificial Ageing of Polymers*, Ed: Th. Reichert, GUS-Publ. No. 5 (2004), ISBN 3-9808382-5-0, pp. 301-318
- [9] VDI 3958-12, *Environmental simulation — Effects of acidic precipitation on polymers — Test methods — Part 12 (Umweltsimulation — Wirkung saurer Niederschläge auf polymere Werkstoffe — Prüfverfahren — Blatt 12)*
- [10] ISO 175, *Plastics — Methods of test for the determination of the effects of immersion in liquid chemicals*
- [11] ISO 1514, *Paints and varnishes — Standard panels for testing*
- [12] ISO 2812-5, *Paints and varnishes — Determination of resistance to liquids — Part 5: Temperature-gradient oven method*
- [13] ISO 2813, *Paints and varnishes — Determination of specular gloss of non-metallic paint films at 20 degrees, 60 degrees and 85 degrees*
- [14] ISO 3668, *Paints and varnishes — Visual comparison of the colour of paints*
- [15] ISO 4628-1, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 1: General introduction and designation system*
- [16] ISO 4628-2, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 2: Assessment of degree of blistering*
- [17] ISO 4628-3, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 3: Assessment of degree of rusting*

- [18] ISO 4628-4, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 4: Assessment of degree of cracking*
- [19] ISO 4628-5, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 5: Assessment of degree of flaking*
- [20] ISO 4628-6, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 6: Assessment of degree of chalking by tape method*
- [21] ISO 4628-7, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 7: Assessment of degree of chalking by velvet method*
- [22] ISO 4628-8, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 8: Assessment of degree of delamination and corrosion around a scribe or other artificial defect*
- [23] ISO 4628-10, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 10: Assessment of degree of filiform corrosion*
- [24] ISO 11664-1, *Colorimetry — Part 1: CIE standard colorimetric observers*
- [25] ISO 11664-2, *Colorimetry — Part 2: CIE standard illuminants*
- [26] ISO 11664-3, *Colorimetry — Part 3: CIE tristimulus values*
- [27] ISO 11664-4, *Colorimetry — Part 4: CIE 1976 L*a*b* Colour space*
- [28] ISO 11664-5, *Colorimetry — Part 5: CIE 1976 L*u*v* Colour space and u', v' uniform chromaticity scale diagram*
- [29] ISO 13803, *Paints and varnishes — Determination of reflection haze on paint films at 20 degrees*
- [30] ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*
- [31] ISO 16474-2, *Paints and varnishes — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*
- [32] ISO 16474-3, *Paints and varnishes — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

