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Paints and varnishes — Artificial weathering and exposure to artificial radiation — Exposure to filtered xenon-arc radiation

Peintures et vernis — Vieillissement artificiel et exposition aux radiations artificielles — Exposition aux radiations filtrées d'une lampe à arc au xénon

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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.

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.

International Standard ISO 11341 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes.*

Annex A forms an integral part of this International Standard. Annex B is for information only.

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Introduction

Coatings of paints, varnishes and similar materials (subsequently referred to simply as coatings) are exposed to artificial weathering, or to artificial radiation, in order to simulate in the laboratory the ageing processes which occur during natural weathering or during exposure tests under glass cover.

In contrast to natural weathering, artificial weathering involves a limited number of variables which can be controlled more readily and which can be intensified to produce accelerated ageing.

The ageing processes which occur during artificial and natural weathering cannot be expected to correlate with each other because of the large number of factors which influence these processes. Definite relationships can only be expected if the important parameters (distribution of the irradiance over the photochemically relevant part of the spectrum, temperature of the specimen, type of wetting and wetting cycle, and relative humidity) are the same in each case or if their effect on the coatings is known.

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Paints and varnishes — Artificial weathering and exposure to artificial radiation — Exposure to filtered xenon-arc radiation

1 Scope

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

It specifies a test method for assessing either the resistance of paint coatings to artificial weathering or the resistance to light by exposure to artificial radiation.

The standard describes the most important parameters and specifies the conditions to be used in the exposure apparatus.

Provided the test conditions specified are strictly respected, reproducibility is improved and an improved correlation is obtained between natural-weathering and artificial-weathering tests.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards

ISO 1512:1991, *Paints and varnishes — Sampling of products in liquid or paste form.*

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1514:1993, *Paints and varnishes — Standard panels for testing.*

ISO 2808:1991, *Paints and varnishes — Determination of film thickness.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4628-1:1982, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 1: General principles and rating schemes.*

ISO 4628-2:1982, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 2: Designation of degree of blistering.*

ISO 4628-3:1982, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 3: Designation of degree of rusting.*

ISO 4628-4:1982, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 4: Designation of degree of cracking.*

ISO 4628-5:1982, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 5: Designation of degree of flaking.*

ISO 4628-6:1990, *Paints and varnishes — Evaluation of degradation of paint coatings — Designation of intensity, quantity and size of common types of defect — Part 6: Rating of degree of chalking by tape method.*

CIE Publication No. 85:1989, *Solar spectral irradiance.*

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 ageing behaviour: The change in properties of a coating during artificial weathering or exposure to artificial radiation until a certain ageing criterion (see 3.3) is satisfied.

NOTE 1 One measure of ageing is the radiant exposure H in the wavelength range below 400 nm or at a specified wavelength, e.g. 340 nm. The ageing behaviour of coatings exposed to artificial weathering, or to artificial radiation, depends on the type of coating, the conditions of exposure of the coating, the property selected for monitoring the progress of the ageing process and the degree of change of this property.

3.2 radiant exposure, H: A measure of the radiant energy to which a test panel has been exposed, given by the formula

$$H = \int E \, dt$$

where

E is the irradiance, in watts per square metre;

t is the exposure time, in seconds.

H is therefore expressed in joules per square metre.

If the irradiance E is constant throughout the whole exposure time, the radiant exposure H is given simply by the product of E and t.

3.3 ageing criterion: A given degree of ageing, corresponding to a specified or agreed degree of change in a selected property of the coating under test.

4 Principle

Artificial weathering of coatings or exposure of coatings to filtered xenon-arc radiation is carried out in order to obtain the degree of change in a selected property after a certain radiant exposure H, and/or the radiant exposure which is required to produce a certain degree of ageing. The properties selected for monitoring should preferably be those which are important for the practical use of the coatings. The properties of the coatings exposed are compared with those of unexposed coatings prepared from the same coating materials at the same time and in the same way (control specimens) or with those of coatings exposed at the same time whose behaviour during testing in exposure apparatus is already known (reference specimens).

During natural weathering, solar radiation is considered to be the essential cause for the ageing of coatings. The same is valid for exposure to radiation under glass. Therefore, in artificial weathering and exposure to artificial radiation, particular importance is attached to the simulation of this parameter. The xenon-arc radiation source used is therefore fitted with one of two different filter systems, designed to modify the spectral distribution of the radiation produced so that, with one of the filters, it matches the spectral distribution, in the ultraviolet and visible regions, of solar radiation (method 1) and, with the other filter, it matches the spectral distribution, in the ultraviolet and visible regions, of solar radiation filtered by 3-mm-thick window-glass (method 2).

Two spectral energy distributions are used to describe the irradiance values and permitted deviations of the filtered test radiation in the ultraviolet range below 400 nm. In addition, CIE Publication No. 85 is used for the specification of the irradiance in the range up to 800 nm because only in that range can the xenon-arc radiation be adapted to match solar radiation sufficiently well.

During testing in exposure apparatus, the irradiance E may change due to ageing of the xenon-arc lamp and the optical-filter system. This occurs particularly in the ultraviolet region which is photochemically important for polymeric materials. Therefore, measurements are made not only of the duration of the exposure, but also of the radiant exposure H in the wavelength range below 400 nm, or at a specific wavelength, e.g. 340 nm, and used as reference values for the ageing of coatings.

It is impossible to simulate accurately every aspect of the way in which the weather acts on coatings. Therefore, in this International Standard, the term artificial weathering is used as distinct from natural weathering. Testing using simulated solar radiation filtered by window-glass is referred to in this International Standard as exposure to artificial radiation.

5 Required supplementary information

For any particular application, the test method specified in this International Standard needs to be completed by supplementary information. The items of supplementary information are given in annex A.

6 Apparatus

6.1 Test chamber

The test chamber shall consist of a conditioned enclosure made from corrosion-resistant material, capable of housing the radiation source, including its filter system, and the test-panel holders.

6.2 Radiation source and filter system

One or more xenon-arc lamps shall be used as the optical radiation source. The radiation emitted by them shall be filtered by a system of optical radiation filters so that the relative spectral distribution of the irradiance (relative spectral energy distribution) in the plane of the test-panel holders is sufficiently similar either to solar ultraviolet and visible radiation (method 1) or to solar ultraviolet and visible radiation filtered by 3-mm-thick window-glass (method 2).

The conditions specified in table 1 shall be used for method 1.

The conditions specified in table 2 shall be used for method 2.

Table 1 — Relative spectral energy distribution for artificial weathering (method 1)

Wavelength, λ nm	Relative irradiance[1)] %
$\lambda < 290$	0
$290 \leqslant \lambda \leqslant 320$	$0,6 \pm 0,2$ [2)]
$320 < \lambda \leqslant 360$	$4,2 \pm 0,5$
$360 < \lambda \leqslant 400$	$6,2 \pm 1,0$
$290 \leqslant \lambda \leqslant 800$	100

1) Relative to the irradiance in the wavelength range from 290 nm to 800 nm (as given in CIE Publication No. 85:1989). See annex B, table B.1.

2) Products that have absorption bands below 300 nm are affected more than under natural weathering conditions if specimens prepared from them are exposed to radiation below 300 nm.

Table 2 — Relative spectral energy distribution for exposure to artificial radiation under window-glass (method 2)

Wavelength, λ nm	Relative irradiance[1)] %
$\lambda < 300$	0
$300 \leqslant \lambda \leqslant 320$	$\leqslant 0,1$
$320 < \lambda \leqslant 360$	$3,0 \pm 0,5$
$360 < \lambda \leqslant 400$	$6,0 \pm 1,0$
$300 \leqslant \lambda \leqslant 800$	100

1) Relative to the irradiance in the wavelength range from 300 nm to 800 nm. See annex B, table B.2.

The radiant flux shall be chosen so that the time-averaged irradiance E between 290 nm and 800 nm in the plane of the test-panel holders is 550 W/m^2. In the case of a discontinuous run (see 9.3), this value shall include the radiation reflected from the inside walls of the test chamber which reaches the plane of the test-panel holders.

The irradiance E at any point over the area used for the test panels shall not vary by more than ± 10 % of the arithmetic mean of the total irradiance for the whole area. Any ozone formed by the operation of the xenon-arc lamps shall not enter the test chamber but shall be vented separately.

In order to accelerate ageing further, deviations from the above specifications concerning relative spectral energy distribution and irradiance may be agreed between the interested parties provided that, for the property selected for the particular coating to be tested, the correlation with natural weathering is known. Such further accelerated-ageing may be carried out either by increasing the irradiance or by shifting the short-wavelength end of the spectral energy distribution band in a defined manner to shorter wavelengths. Details of any such deviations from the methods specified shall be stated in the test report.

Ageing of the xenon-arc lamps and filters causes the relative spectral energy distribution to change during operation and the irradiance to decrease. Renewal of the lamps and filters will keep the spectral energy distribution and the irradiance constant. The irradiance may also be kept constant by adjustment of the apparatus. Follow the manufacturer's instructions.

6.3 Test-chamber conditioning system

To maintain the test chamber at the black-standard temperature specified in 9.2, humidity- and temperature-controlled dust-free air shall be circulated through the test chamber. The temperature and relative humidity of the air in the test chamber shall be monitored using temperature and humidity sensors protected against direct radiation. To maintain the relative humidity at the level specified in 9.4, only distilled or demineralized water shall be used.

NOTE 2 When the test chamber is fed continuously with fresh air, the operating conditions of the apparatus may differ, for example, in the summer from those in the winter, because the moisture content of the air in the summer is higher than in the winter. This may influence the test results. The reproducibility of the test may be improved by circulating the air in an essentially closed circuit.

6.4 Device for wetting the test panels (for use in method 1)

NOTE 3 Method 1 includes wetting of the test panels; this is intended to simulate the effects of rain and condensation in an outdoor environment.

The device for wetting the test panels shall be designed so that, during the whole of the wetting period specified in 9.4, the surface under test of all the test panels shall be wetted in one of the following ways:

a) the surface is sprayed with water;

b) the test chamber is flooded with water.

If the test panels rotate around the radiation source, the water-spray nozzles shall be arranged so that the requirements of 9.4 are met for each test panel.

Distilled or demineralized water meeting the requirements of ISO 3696, grade 2, having a conductivity below 2 µS/cm but a residue on evaporation of less than 1 ppm, shall be used for wetting.

Recycled water shall not be used unless filtered to give water of ISO 3696 grade 2 purity since there is a danger of deposits forming on the test-panel surfaces. Such deposits may lead to false results.

The supply tanks, supply pipes and spray nozzles for the distilled or demineralized water shall be made of corrosion-resistant material.

6.5 Test-panel holders

The holders for the test panels shall be made of an inert material.

6.6 Black-standard thermometer

The black-standard thermometer for measuring the black-standard temperature in the plane of the test panels during the dry period shall consist of a plane stainless steel plate with a thickness of about 0,5 mm, a length of about 70 mm and a width of about 40 mm. The surface of this plate facing the radiation source shall be coated with a flat black layer with good resistance to ageing. The black surface shall absorb at least 93 % of all incident radiation up to 2 500 nm. The temperature of the plate shall be measured with an electrical sensor fitted in good thermal contact with the centre of the plate on the side not exposed to the radiation source. This side of the metal plate shall be fixed to a 5-mm-thick backing plate made of unfilled poly(vinylidene fluoride) (PVDF), leaving a closed air space in the area of the sensor. The distance between the sensor and the recess in the PVDF plate shall be about 1 mm. The length and the width of the PVDF plate shall be sufficiently large to ensure that, when fitting the black-standard thermometer to a test-panel holder, no metal-to-metal thermal contact exists between the metal plate and the test-panel holder and that the metal mounts of the holder are at least 4 mm from the edges of the metal plate.

If any change in appearance of the black surface is observed, follow the manufacturer's instructions.

NOTES

4 The black-standard thermometer described differs from the black-panel thermometer formerly used in that the black panel is mounted in a thermally insulated mounting. The temperatures measured therefore correspond approximately to those measured on the exposed surface of test-panels with a black or dark-coloured coating on a substrate of low thermal conductivity. The surface temperatures of light-coloured test panels will usually be lower values.

5 The surface temperature of a test panel depends on a number of factors, including the amount of radiation absorbed, the amount of radiation emitted, thermal-conduction effects within the test panel and heat transfer between the test panel and the air and between the test panel and the holder, and cannot therefore be predicted with any accuracy.

In order to be able to determine the range of surface temperatures of the test panels during exposure, and to control better the exposure conditions in the apparatus, the use of an analogously designed white-standard thermometer is recommended in addition to the black-standard thermometer. For this purpose, a white coating with good ageing resistance having a reflectance of at least 90 % in the wavelength range between 300 nm and 1 000 nm and at least 60 % between 1 000 nm and 2 000 nm shall be used.

6.7 Radiometer

The irradiance E and radiant exposure H of the surfaces of the test panels in the test chamber shall be measured using a radiometer with a photo-electric receiver cell with a field of view of 2π sr and a good cosine response. The radiometer shall be calibrated on the basis of the spectral distribution given in table B.1. The calibration shall be checked in accordance with the manufacturer's instructions.

NOTE 6 Direct comparison of the radiant exposure measured in the exposure apparatus with that measured during natural weathering is possible if the radiometer used is of the same type in each case.

7 Sampling

Take a representative sample of the product to be tested (or of each product in the case of a multi-coat system), as described in ISO 1512.

Examine and prepare each sample for testing, as described in ISO 1513.

8 Preparation of test panels

The substrate used for the preparation of the test panels shall be that usually used in practice (e.g. plasterboard, wood, metal, plastic materials) and the method of application and drying of the coating shall be that normally used in practice to give a coating of the usual thickness.

Unless otherwise agreed or specified, standard panels in accordance with ISO 1514 shall be used as substrate for the test coating.

NOTE 7 Preferably, flat test panels of dimensions appropriate to the holders in the test chamber should be used.

Unless otherwise agreed, only the front sides of the test panels shall be coated with the coating material or coating system to be tested. The rear sides and the edges of the test panels shall be coated, if necessary, with a coating of protective paint of sufficient mechanical strength.

Stoving paints shall be dried under the same conditions as laid down for their normal use. In the case of air-drying paints, the coated test panels shall be stored horizontally and allowed to dry at a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %. The duration of drying and subsequent storage shall be as specified.

All the test panels shall be permanently marked in a suitable way. The thickness of the test coating shall be determined in accordance with ISO 2808.

In the case of testing carried out over a series of different test periods, an adequate number of test panels shall be prepared for each coating material.

If required, at least one additional test panel for each coating shall be prepared, and stored at a temperature of 18 °C to 28 °C in the dark for use as a control specimen. It shall be borne in mind, however, that such specimens may change their properties during storage.

Coatings like alkyd paints which are sensitive to storage in the dark shall be stored under conditions agreed between the interested parties.

9 Procedure

9.1 Mounting the test panels

Mount the test panels in the holders (6.5) so that the surrounding atmosphere has free access to the coatings to be tested.

NOTE 8 It may be agreed that the arrangement of the test panels in the holders be changed at regular intervals, for example the upper row exchanged with the lower row.

9.2 Black-standard temperature

For normal testing, set the black-standard temperature to (65 ± 2) °C. If the test panels are periodically wetted during exposure, measure the black-standard temperature at the end of each dry phase. Even when the apparatus is being operated in the discontinuous mode, always use the black-standard thermometer (6.6) continuously (see 9.3).

When testing for colour changes, use a black-standard temperature of (55 ± 2) °C. At higher temperatures, large-scale degradation of the binder occurs, leading to chalking and loss of gloss, making the accurate assessment of colour changes difficult.

9.3 Exposure of test panels

Expose the test panels, and the radiometer (6.7) if mounted on the rack, either continuously (continuous run) or with periodically changing irradiance (discontinous run), using method 1 or method 2 (see clause 4) and operating the black-standard thermometer continuously in either case.

If operating in the discontinuous mode, the periodic changes in irradiance are produced by rotating the test panel holders through an angle of 180° to turn the test panels out of and bring them back into the direct radiation from the radiation source.

NOTE 9 Discontinuous operation may be necessary in order to ensure that the mean irradiance is that specified in 6.2.

9.4 Wetting the test panels and relative humidity in the test chamber

Unless otherwise agreed, wet the test panels repeatedly as specified for cycles A and B or keep the relative humidity in the test chamber constant as specified for cycles C and D (see table 3).

Table 3 — Test panel wetting cycles

Cycle	A	B	C	D
Operating mode	Continuous run	Discontinuous run	Continuous run	Discontinuous run
Wetting time, min	18	18	—	—
Drying period, min	102	102	permanent	permanent
Relative humidity during drying period, %	60 to 80	60 to 80	40 to 60	40 to 60