INTERNATIONAL STANDARD

ISO 4892-1

Second edition 1999-07-01

Plastics — Methods of exposure to laboratory light sources —

Part 1:

General guidance

Plastiques — Méthodes d'exposition à des sources lumineuses de laboratoire —

Partie 1: Guide général





1999年9月10日

Reference number ISO 4892-1:1999(E)

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ISO 4892-1:1999(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 3.

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 4892-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 4892-1:1994), of which it constitutes a technical revision.

ISO 4892 consists of the following parts, under the general title Plastics — Methods of explosure to laboratory light sources:

- Part 1: General guidance
- Part 2: Xenon-arc sources
- Part 3: Fluorescent UV lamps
- Part 4: Open-flame carbon-arc lamps

Annex B forms a normative part of this part of ISO 4892. Annex A is for information only.

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Introduction

Plastics are often used outdoors or in indoor locations where they are exposed to daylight or to daylight behind glass for long periods. It is therefore very important to determine the effects of daylight, heat, moisture and other climatic stresses on the colour and other properties of plastics. Outdoor exposures to daylight and to daylight filtered by window glass are described in ISO 877:1994, Plastics — Methods of exposure to direct weathering, to weathering using glass-filtered daylight, and to intensified weathering by daylight using Fresnel mirrors. However, it is often necessary to determine more rapidly the effects of light, heat and moisture on the physical, chemical and optical properties of plastics with accelerated laboratory exposure tests that use specific laboratory light sources. Exposures in these laboratory devices are conducted under more controlled conditions than found in natural environments and are designed to accelerate polymer degradation and product failures.

Relating results from accelerated laboratory exposures to those obtained in actual-use conditions is difficult because of variability in both types of exposure and because laboratory tests often do not reproduce all the exposure stresses experienced by plastics exposed in actual-use conditions. No single laboratory exposure test can be specified as a total simulation of actual-use exposures.

The relative durability of materials in actual-use exposures can be very different depending on the location of the exposure because of differences in UV radiation, time of wetness, temperature, pollutants and other factors. Therefore, even if results from a specific accelerated laboratory test are found to be useful for comparing the relative durability of materials exposed in a particular outdoor location or in particular actual-use conditions, it cannot be assumed that they will be useful for determining the relative durability of materials exposed in a different outdoor location or in different actual-use conditions.

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Plastics — Methods of exposure to laboratory light sources —

Part 1:

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General guidance

1 Scope

- 1.1 This part of ISO 4892 provides information and general guidance relevant to the selection and operation of the methods of exposure described in detail in subsequent parts. It also describes and recommends procedures for determining irradiance and radiant exposure. Requirements for devices used to monitor chamber air temperature and surface temperature of dark and light materials are also described.
- 1.2 This part of ISO 4892 also provides information on the interpretation of data from accelerated exposure tests. More specific information about methods for determining the change in plastic properties after exposure and reporting these results is described in ISO 4582.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 4892. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 4892 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, Plastics — Standard atmospheres for conditioning and testing.

ISO 293:1986, Plastics — Compression moulding test specimens of thermoplastic materials.

ISO 294-1:1996, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens.

ISO 294-2:1996, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars.

ISO 294-3:1996, Plastics -- Injection moulding of test specimens of thermoplastic materials -- Part 3: Small plates.

ISO 295:1991, Plastics — Compression moulding of test specimens of thermosetting materials.

ISO 2557-1:1989, Plastics — Amorphous plastics — Preparation of test specimens with a specified maximum reversion — Part 1: Bars.

ISO 2818:1994, Plastics - Preparation of test specimens by machining.

ISO 3167:1993, Plastics — Multipurpose test specimens.

ISO 4582:1998, Plastics — Determination of changes in colour and variations in properties after exposure to daylight under glass, natural weathering or laboratory light sources.

ISO 4892-2:1994, Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc sources.

ISO 4892-3:1994, Plastics — Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps.

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ISO 4892-4:1994, Plastics — Methods of exposure to laboratory light sources — Part 4: Open-flame carbon-arc lamps.

ISO 9370:1997, Plastics — Instrumental determination of radiant exposure in weathering tests — General guidance and basic test method.

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

3 Terms and definitions

For the purposes of this part of ISO 4892, the following terms and definitions apply.

3.1

centrol

(weathering) a material which is of similar composition and construction to the test material and which is exposed at the same time for comparison with the test material

NOTE An example of the use of a control material would be when a formulation different from one currently being used is being evaluated. In that case, the control would be the plastic made with the original formulation.

3.2

file specimen

a portion of the material to be tested which is stored under conditions in which it is stable and is used for comparison between exposed and original state

3.3

reference material

a material of known performance

3.4

reference specimen

a portion of the reference material that is to be exposed

4 Principle

Specimens of the samples to be tested are exposed to laboratory light sources under controlled environmental conditions. The methods described include means which may be used to measure irradiance at the face of the specimen and radiant exposure, and procedures for measuring the temperature of specified white and black panels.

4.1 Significance

- **4.1.1** When conducting exposures in devices which use laboratory light sources, it is important to consider how well the accelerated-test conditions simulate the actual-use environment for the plastic being tested. In addition, it is essential to consider the effects of variability in both the accelerated test and actual exposures when setting up exposure experiments, and when interpreting the results from accelerated exposure tests.
- 4.1.2 No laboratory exposure test can be specified as a total simulation of actual-use conditions. Results obtained from these laboratory accelerated exposures can be considered as representative of actual-use exposures only when the degree of rank correlation has been established for the specific materials being tested and when the type and mechanism of degradation are the same. The relative durability of materials in actual-use conditions can be very different locations because of differences in UV radiation, time of wetness, relative humidity, temperature, pollutants and other factors. Therefore, even if results from a specific exposure test conducted in accordance with ISO 4892 are found to be useful for comparing the relative durability of materials exposed in a particular environment, it cannot be assumed that they will be useful for determining the relative durability of the same materials in a different environment.
- **4.1.3** Even though it is very tempting, calculation of an "acceleration factor" relating "x" hours or megajoules of radiant exposure in an accelerated laboratory test to "y" months or years of actual exposure is *not recommended*. These acceleration factors are not valid for several reasons.

- a) Acceleration factors are material-dependent and can be significantly different for each material and for different formulations of the same material.
- b) Variability in the rate of degradation in both actual-use and accelerated laboratory exposure tests can have a significant effect on the calculated acceleration factor.
- c) Acceleration factors calculated based on the ratio of irradiance between a laboratory light source and daylight (even when identical bandpasses are used) do not take into consideration the effects of temperature, moisture and differences in spectral power distribution between the laboratory light source and daylight.
- NOTE If use of an acceleration factor is desired in spite of the warnings given in this standard, such acceleration factors for a particular material are only valid if they are based on data from a sufficient number of separate exterior or indoor environmental tests and accelerated laboratory exposures so that results used to relate times to failure in each exposure can be analysed using statistical methods. An example of a statistical analysis using multiple laboratory and actual exposures to calculate an acceleration factor is described by J.A. Simms, in *Journal of Coatings Technology*, Volume 50, 1987, pages 45-53.
- 4.1.4 There are a number of factors that may decrease the degree of correlation between accelerated tests using laboratory light sources and exterior exposures (more specific information on how each factor may after the stability ranking of materials is given in annex A):
- a) differences in the spectral distribution of the laboratory light source and daylight;
- b) light intensities higher than those experienced in actual-use conditions;
- c) exposure cycles that use continuous exposure to light from a laboratory light source without any dark periods;
- d) specimen temperatures higher than those in actual conditions;
- e) exposure conditions that produce unrealistic temperature differences between light- and dark-coloured specimens;
- f) exposure conditions that produce very frequent cycling between high and low specimen temperatures, or that produce unrealistic thermal shock;
- g) unrealistically high or low levels of moisture;
- h) the absence of biological agents or pollutants.

4.2 Use of accelerated tests with laboratory light sources

- 4.2.1 Results from accelerated exposure tests conducted in accordance with this standard are best used to compare the relative performance of materials. A common application of this is tests conducted to establish that the level of quality of different batches does not vary from that of a control material of known performance. Comparisons between materials are best made when the materials are tested at the same time in the same exposure device. Results can be expressed by comparing the exposure time or radiant exposure necessary to reduce the level of a characteristic property to some specified level.
- **4.2.1.1** It is strongly recommended that at least one control material be exposed with each test for the purpose of comparing the performance of the test materials to that of the control. The control material should be of similar composition and construction and be chosen so that its failure modes are the same as that of the material being tested. It is preferable to use two controls, one with relatively good durability and one with relatively poor durability.
- **4.2.1.2** Sufficient replicates of each control material and each test material being evaluated are necessary in order to allow statistical evaluation of the results. Unless otherwise specified, use a minimum of three replicates for all test and control materials. When material properties are measured using destructive tests, a separate set of specimens is needed for each exposure period.
- 4.2.2 In some specification tests, test materials are exposed at the same time as a weathering reference material (e.g. blue wool test fabric). The property or properties of the test material are measured after a defined property of the reference material reaches a specified level. If the reference material differs in composition from the test material, it may not be sensitive to exposure stresses which produce failure in the test material, or it may be very sensitive to an exposure stress that has very little effect on the test material. The variability in results for the reference material may be much different than that for the test material. All these differences between the reference material and the test material can produce misleading results.

NOTE Definitions of control and reference material that are appropriate to weathering tests are given in clause 3.

4.2.3 In some specification tests, properties of test specimens are evaluated after a specific exposure time or radiant exposure using a test cycle with a prescribed set of conditions. Results from any accelerated exposure test conducted in accordance with this standard should not be used to establish a "pass/fail" of materials based on the level of a specific property after a specific exposure time or radiant exposure unless the reproducibility of the effects of a particular exposure cycle and property measurement method has been established.

5 Requirements for laboratory exposure devices

5.1 Light source

- **5.1.1** The exposure device shall provide for placement of specimens and any designated sensing devices in positions that allow uniform irradiance from the light source.
- **5.1.2** Exposure devices shall be designed such that the irradiance at any location in the area used for specimen exposures is at least 70 % of the maximum irradiance measured in this area. Procedures for measuring irradiance uniformity are found in annex B.
- NOTE The irradiance uniformity in exposure devices depends on several factors such as deposits which can develop on the optical system and chamber walls. In addition, irradiance uniformity can be affected by the type and number of specimens being exposed. The irradiance uniformity as guaranteed by the manufacturer is valid for new equipment and well defined measuring conditions. In many new-model devices, the irradiance at any location within the exposure area is at least 80 % of the maximum irradiance.
- **5.1.3** If the irradiance at any position in the area used for specimen exposure is at least 90 % of the maximum irradiance, periodic repositioning of the specimens during exposure is not necessary.
- NOTE While not required in devices meeting the irradiance uniformity requirements of 5.1.3, periodic specimen repositioning is a good practice to ensure that all specimens receive the same level of all exposure stresses.
- **5.1.4** If irradiance at any position in the area used for specimen exposure is between 70 % and 90 % of the maximum irradiance, specimens shall be periodically repositioned during the exposure period to ensure that each receives an equal amount of radiant exposure. The repositioning schedule shall be agreed upon by all interested parties.
- **5.1.5** Follow the apparatus manufacturer's instructions for lamp and filter replacement and for pre-ageing of lamps and/or filters.
- **5.1.6** CIE Publication No. 85:1989 provides data on solar spectral irradiance for typical atmospheric conditions, which can be used as a basis for comparing laboratory light sources with daylight. For example, global solar irradiance in the 300 nm to 2 450 nm band is given as 1 090 W/m² for a relative air mass of 1, with 1,42 cm of precipitable water and 0,34 cm of ozone (measured at a pressure of 1 atmosphere and a temperature of 0 °C). Table 1 shows a broadband condensed spectral irradiance for global solar radiation at these atmospheric conditions in the UV, visible and infrared regions of the spectrum. This represents the maximum global solar irradiance that would be experienced by materials exposed on a horizontal surface at the Equator near noon on a clear day at the spring or autumn equinox.

Table 1 — Spectral global irradiance (condensed from Table 4 of CIE Publication No. 85:1989)

Wavelength	Irradiance	Percent of total	Percent of UV and visible
nm	W·m ⁻²	300 nm to 2 450 nm	300 nm to 800 nm
300 to 320 320 to 360 360 to 400 300 to 400 400 to 800 300 to 800 800 to 2 450 300 to 2 450	4,1 28,5 42,0 74,6 604,2 678,8 411,6 1090,4	0,4 2,6 3,9 6,8 55,4 62,2 37,8 100,0	0,6 4,2 6,2 11,0 89,0 100,0

- 5.1.6.1 Direct radiation from xenon burners, open-flame carbon arcs and some fluorescent lamps contains considerable amounts of short-wavelength ultraviolet radiation not present in daylight. With proper selection of filters for these light sources, much of the short-wavelength light can be eliminated. However, some filters allow a small, but significant, amount of this short-wavelength (less than 300 nm) radiation through. Fluorescent lamps can be selected to have a spectral output corresponding to a particular ultraviolet region of daylight. The xenon arc, when appropriately filtered, produces radiation with a spectral power distribution that is a good simulation of average daylight throughout the UV and visible region.
- **5.1.7** A radiometer which complies with the requirements outlined in ISO 9370 may be used to measure the irradiance E or spectral irradiance E_{λ} and the radiant exposure H or spectral radiant exposure H_{λ} on the specimen surface
- **5.1.7.1** If used, the radiometer shall be mounted so that it receives the same radiation as the specimen surface. If it is not positioned in the specimen plane, it shall have a sufficient field of view and be calibrated for irradiance at the specimen distance.
- 5.1.7.2 The radiometer shall be calibrated in the emission region of the light source used. Calibration shall be checked in accordance with the radiation measuring instrument manufacturer's instructions. A full calibration of the radiometer shall be conducted at least once per year by an approved, accredited laboratory. More frequent calibrations are recommended.
- **5.1.7.3** When measured, the irradiance in the wavelength range agreed upon by all interested parties shall be reported. Some types of apparatus provide for measuring irradiance in a specific wavelength range (e.g. 300 nm to 400 nm or 300 nm to 800 nm), or in a narrow bandpass centered around a single wavelength (e.g. 340 nm).

5.2 Temperature

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- **5.2.1** The surface temperature of exposed materials depends primarily on the amount of radiation absorbed, the emissivity of the specimen, the amount of thermal conduction within the specimen and the amount of heat transmission between the specimen and the air or between the specimen and the specimen holder. Since it is not practical to monitor the surface temperature of individual test specimens, a specified black-panel sensor is used to measure and control the temperature within the test chamber. It is strongly recommended that the black-panel temperature sensor be mounted on a support within the specimen exposure area so that it receives the same radiation and experiences the same cooling conditions as a flat test panel surface using the same support. The black panel may also be located at a fixed distance from the light source different from that of the test specimens and calibrated to give the temperature in the specimen exposure area. However, this is not recommended because a black panel mounted at a fixed position away from the specimens may not indicate temperatures representative of the test specimens, even if it is calibrated to record the temperature at positions within the specimen exposure area, due to differences in light intensity and movement of air.
- **5.2.2** Two types of black-panel temperature sensor may be used:
- **5.2.2.1** Black-standard thermometers, consisting of a plane (flat) stainless-steel plate with a thickness of about 0,5 mm. A typical length and width is about 70 mm by 40 mm. The surface of this plate facing the light source shall be coated with a black layer which has good resistance to ageing. The coated black plate shall absorb at least 90 % to 95 % of all incident flux to 2500 nm. A platinum resistance sensor shall be attached in good thermal contact to the centre of the plate on the side opposite the radiation source. This side of the metal plate shall be attached to a

5 mm thick baseplate made of unfilled poly(vinylidene fluoride) (PVDF). A small space sufficient to hold the platinum resistance sensor shall be machined in the PVDF baseplate. The distance between the sensor and this recess in the PVDF plate shall be about 1 mm. The length and width of the PVDF plate shall be sufficient so that no metal-to-metal thermal contact exists between the black-coated metal plate and the mounting holder into which it is fitted. The metal mounts of the holder of the insulated black panel shall be at least 4 mm from the edges of the metal plate. Black-standard thermometers which differ in construction are permitted as long as the temperature indicated by the alternative construction is within \pm 1,0 °C of that of the specified construction at all steady-state temperature and irradiance settings the exposure device is capable of attaining. In addition, the time needed for an alternative black-standard thermometer to reach the steady state shall be within 10 % of the time needed for the specified black-standard thermometer to reach the steady state.

- 5.2.2.2 Black-panel thermometers, consisting of a plane (flat) metal plate that is resistant to corrosion. Typical dimensions are about 150 mm long, 70 mm wide, and 1 mm thick. The surface of this plate that faces the light source shall be coated with a black layer which has good resistance to ageing. The coated black plate shall absorb at least 90 % to 95 % of all incident flux to 2500 nm. A thermally sensitive element shall be firmly attached to the centre of the exposed surface. This thermal sensitive element can be a black-coated stem-type bimetallic dial sensor or a resistance sensor. The back of the metal panel shall be open to the atmosphere within the exposure chamber.
- 5.2.3 The temperature indicated by the black-panel or black-standard thermometer depends on the irradiance produced by the laboratory light source and the temperature and speed of the air moving in the test chamber. Black-panel temperatures generally correspond to those for dark coatings on metal panels. Black-standard thermometer temperatures generally correspond to those for the exposed surface of dark samples with poor thermal conductivity. At conditions used in typical exposure tests, the temperature indicated by a black-standard thermometer will be 3 °C to 12 °C higher than that indicated by a black-panel thermometer. Because black-standard thermometers are insulated, their response time for temperature changes is slightly slower than for a black-panel thermometer.
- 5.2.3.1 At low irradiance levels, the difference between the temperature indicated by a black-panel or black-standard thermometer and the real specimen temperature may be small. When light sources that emit very little infrared radiation are used, there will generally be only very small differences in the temperatures indicated by the two types of black panel or between light- and dark-coloured specimens.
- **5.2.4** In order to evaluate the range of surface temperatures of the exposed specimens and to better control the irradiance or the conditions in the apparatus, the use of a white-panel or white-standard thermometer is recommended, in addition to the black-panel or black-standard thermometer. The white-panel or white-standard thermometer shall be constructed in the same way as the corresponding black-panel or black-standard thermometer, except for use of a white coating with a good resistance to ageing. The reflectance of the white coating between 300 nm and 1 000 nm shall be at least 90 %, and at least 60 % between 1 000 and 2 500 nm.
- 5.2.5 Exposure devices shall be able to control the temperature of the black or white temperature sensor to within ± 3 °C of the desired temperature. They shall be designed such that the temperature of a black- or white-panel temperature sensor placed anywhere within the specimen exposure area is within ± 5 % of the desired Celsius temperature.
- **5.2.6** The test report shall indicate whether a black-standard or black-panel thermometer was used. If any thermometer used (black or white) is not positioned in the specimen exposure area, its exact position shall be described in the test report.

5.3 Humidity and wetting

- 5.3.1 The presence of moisture, particularly in the form of condensation on the exposed face of the specimen, may have a significant effect in accelerated laboratory exposure tests. Any apparatus operated in accordance with this standard which attempts to simulate the effects of moisture shall have means for providing moisture to specimens using one or more of the following methods:
- a) humidification of the chamber air;
- b) formation of condensation;
- c) water spray;
- d) immersion.

- 5.3.2 The purity of the water used for spraying the specimens is very important. Without proper treatment to remove cations, anions, organics and, in particular, silica, exposed specimens will develop spots or stains that do not occur in exterior exposures. It is strongly recommended that water used for specimen spray contain a maximum of 1 µg/g of solids and a maximum of 0.2 µg/g of silica. Distillation, or a combination of deionization and reverse osmosis, can effectively produce water of the desired purity. If the water used for specimen spray contains more than 1 µg/g of solids, the solids and silica levels shall be reported. Recirculation of water used for specimen spray is not recommended and shall not be done unless the recirculated water meets the purity requirements listed above.
- **5.3.3** If specimens are found to have deposits or stains after exposure, the water purity shall be checked to determine if it meets the purity requirements described in 5.3.2. On some occasions, exposed specimens can be contaminated by deposits from bacteria that can grow in the purified water used for specimen spray. If bacterial contamination is detected, the entire system used for specimen water spray shall be flushed with a chlorinating solution such as sodium hypochiorite and thoroughly rinsed prior to resuming exposures.
- 5.3.4 Although conductivity does not always correlate with silica content, it is recommended that the conductivity of the water used for specimen spray be continuously monitored and that exposures be stopped whenever the conductivity is above 5 µS/cm.
- **5.3.5** All components of the specimen spray unit shall be fabricated from stainless steel, or some other material that does not contaminate the water with materials that could absorb UV radiation or form unrealistic deposits on test specimens.

5.4 Other apparatus requirements

- **5.4.1** Although various apparatus designs are used in practice, each apparatus shall meet the following requirements:
- **5.4.1.1** In devices where humidity within the test chamber is controlled, the sensors used to measure the humidity shall be placed within the test chamber air flow and shielded from direct radiation and water spray.
- **5.4.1.2** In devices where chamber air temperature is controlled, temperature sensors, shielded from direct radiation and water spray, are required to sense and control the temperature of the air within the test chamber.
- **5.4.1.3** Any device intended to simulate the effects of moisture shall have means to programme intervals with and without wetting of the specimens.
- **5.4.1.4** Any device intended to simulate the effects of light and dark cycles shall have an electronic controller or mechanical device to programme intervals with or without radiation.
- **5.4.2** To fulfill the requirements of particular test procedures, the apparatus may need to provide means to register or record the following operating parameters:
- a) the line voltage;
- b) the lamp voltage;
- c) the lamp current;
- the temperature indicated by any uninsulated or insulated black- or white-panel thermometer;
- e) the test chamber air temperature;
- f) the relative humidity of the test chamber;
- g) the details of any water spray cycle;
- the spectral irradiance and the radiant exposure;
- i) the duration of exposure (radiation time and total, if different).



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6 Test specimens

6.1 Form, shape and preparation

- **6.1.1** The methods used for the preparation of test specimens can have a significant impact on their apparent durability. Therefore, the method used for specimen preparation shall be agreed upon by the interested parties. It should preferably be closely related to the method normally used to process the material in typical applications. A complete description of the method used for the preparation of test specimens shall be included with the test report.
- **6.1.2** The dimensions of the test specimens are normally those specified in the appropriate test method for the property or properties to be measured after exposure. When the behaviour of a specific type of article is to be determined, the article itself should be exposed whenever possible.
- 6.1.3 If the material to be tested is an extrusion- or moulding-grade polymer in the form of granules, chips, pellets or some other raw state, specimens to be exposed shall be cut from a sheet produced by the appropriate method. The exact shape and dimensions of the specimens will be determined by the specific test procedure used for measurement of the property of interest. The procedures used to machine or cut individual test specimens from a larger sheet or article may affect the results of the property measurement, and hence the apparent durability of the specimens. For preparation of test specimens, the procedures described in ISO 293, ISO 294-1, -2 and -3, ISO 295, ISO 2557-1 and ISO 3167 have been found to be satisfactory.
- **6.1.4** In some cases, individual specimens used for property measurement may need to be cut from a larger specimen which has been exposed. For example, materials that delaminate at the edges may be exposed in the form of larger sheets from which individual test specimens are cut after exposure. The effects of any cutting or machining operation on the properties of individual test specimens are usually much larger when the test specimens are cut from a large piece after exposure. This is especially true for materials that embrittle on exposure. Follow the procedures described in ISO 2818 for preparation of test specimens by machining. Only cut individual test specimens for property measurement from larger specimens that have been exposed when this preparation procedure is specifically specified.

When test specimens are cut from an exposed sheet or larger article, they should preferably be taken from an area that is at least 20 mm from the fixture holding the material or from the exposed specimen edges. In no circumstances shall any material from the exposed face be removed during test specimen preparation.

6.1.5 When comparing materials in an exposure test, use test specimens that are similar in dimensions and exposed area.

6.2 Number of test specimens

- **6.2.1** The number of test specimens for each set of test conditions or exposure period shall be that specified in the appropriate test method for the property or properties to be measured after exposure.
- NOTE For the determination of mechanical properties, it is recommended that the number of test specimens exposed be twice that required by the relevant International Standard (due to the large standard deviation known to occur in measuring the mechanical properties of "weathered" materials).
- **6.2.2** If the test method used for property measurement does not specify the number of test specimens to be exposed, it is recommended that a minimum of three replicate specimens of each material be prepared for each exposure stage.
- **6.2.3** When destructive tests are used to determine the properties being measured, the total number of test specimens required will be determined by the number of exposure periods used and whether unexposed file specimens are tested at the same time as exposed specimens.
- **6.2.4** Control materials of known durability should preferably be included with each exposure test. It is recommended that control materials known to have relatively poor and relatively good durability be used. Before any laboratory to laboratory comparisons are made, it is necessary for all the interested parties to agree on the control materials to be used. The number of specimens of the control material should preferably be the same as that used for test materials.

6.3 Storage and conditioning

- **6.3.1** If test and/or reference specimens are cut or machined from larger pieces, they shall be conditioned, after preparation, in accordance with ISO 291. In some circumstances, it may be necessary to precondition the sheets prior to cutting or machining to facilitate specimen preparation.
- 6.3.2 When using tests to characterize the mechanical properties of the materials being exposed, specimens shall be appropriately conditioned before all property measurements. Use the conditions described in ISO 291, where appropriate. The properties of some plastics are very sensitive to moisture content, and the duration of conditioning may need to be longer than that specified in ISO 291, particularly where specimens have been exposed to climatic extremes.
- **6.3.3** File specimens shall be stored in the dark under normal laboratory conditions, preferably in one of the standard atmospheres given in ISO 291.
- **6.3.4** Some materials will change colour during storage in the dark, particularly after weathering. It is essential that colour measurement or visual comparison be carried out as soon as possible after exposure once the exposed surface has dried.

7 Test conditions and procedure

- 7.1 The conditions and procedures for the exposure test depend upon the particular method selected. Refer directly to the appropriate part (part 2, 3 or 4) of ISO 4892.
- 7.2 Follow the procedures described in ISO 4582 for measuring properties of test specimens before and after exposure and for expressing the change in properties after exposure. Refer to the relevant International Standard for the specific procedures to follow for measuring specimen properties.
- 7.2.1 If non-destructive tests are used to measure properties of the materials being tested, the properties of the test specimens shall be measured before beginning the exposure. The same property is then measured after each exposure period. Care shall be taken to make the property measurement after each exposure period in the same position on the test specimen.
- NOTE To monitor the response of the instrument used to measure the desired property, one can carry out a measurement on a reference or calibration specimen each time the test instrument is used.
- 7.2.2 If destructive tests are used to measure properties of the materials being tested, separate sets of test specimens will be needed for each exposure period. The property is measured on each set of exposed specimens. The value of the property after exposure may be compared to that obtained prior to exposure. Alternatively, the property can be measured on a separate set of file specimens at the same time as the property of the exposed specimens is measured. The results for the file specimens and from the exposed specimens can then be compared.

8 Precision and bias

8.1 Precision

- **8.1.1** The repeatability and reproducibility of results obtained in exposures conducted in accordance with this standard will vary with the materials being tested, the material property being measured, and the specific test conditions and cycles that are used. In round-robin studies¹⁾ conducted by ASTM subcommittee G3.03, the 60° gloss values of replicate PVC tape specimens exposed in different laboratories using identical test devices and exposure cycles showed significant variability. The variability shown in these round-robin studies restricts the use of "absolute specifications" such as requiring a specific property level after a specific exposure period.
- **8.1.1.1** If a standard or specification for general use requires a definite property level after a specific time or radiant exposure in an exposure test conducted in accordance with this practice, the specified property level shall be based

¹⁾ Fischer, R.M., "Results of Round-Robin Studies of Light- and Water-Exposure Standard Practices", Accelerated and Outdoor Durability Testing of Organic Materials, ASTM STP 1202, Warren D Ketola and Douglass Grossman (editors), American Society for Testing and Materials, Philadelphia, 1993.

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on results obtained in a round-robin that takes into consideration the variability due to the exposure and the test method used to measure the property of interest. The round-robin shall be conducted in accordance with the relevant International Standard for conducting round-robin exposures and shall include a statistically representative sample of all laboratories or organizations which would normally conduct the exposure and property measurement.

- **8.1.1.2** If a standard or specification for use between two or three parties requires a definite property level after a specific time or radiant exposure in an exposure test conducted in accordance with this practice, the specified property level shall be based on statistical analysis of results from at least two separate, independent exposures in each laboratory. The design of the experiment used to determine the specification shall take into consideration the variability due to the exposure and the test method used to measure the property of interest.
- 8.1.2 The same round-robin studies cited in 8.1.1 demonstrated that the gloss values for a series of materials could be ranked with a high level of reproducibility between laboratories. When reproducibility in results from an exposure test conducted in accordance with this standard have not been established through round-robin testing, performance requirements for materials shall be specified in terms of comparison (i.e. rank) with a control material. Specimens of the control material shall be exposed simultaneously with the test specimen(s) in the same device. The specific control material used shall be agreed upon by the interested parties.

8.2 Bias

Bias cannot be determined because no acceptable standard weathering reference materials are available.

9 Test report

The test report shall contain the following information:

- 9.1 Specimen description
- a) A full description of the specimens and their origin.
- b) Compound details, cure time and temperature where appropriate.
- c) 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.

- 9.2 Description of exposure test conducted in accordance with part 2, 3 or 4 of ISO 4892, including:
- a) A description of the exposure device and light source, including:
 - 1) the type of device and light source;
 - 2) a description of the filters used;
 - 3) if required, the irradiance at the specimen surface (including the bandpass in which the radiation was measured);
 - 4) the number of hours that the filters and the light source had been used prior to beginning the exposure.
- b) The type of black- and/or white-panel temperature sensor used and the exact position of the sensor if it was not located in the test specimen exposure area.
- c) If required, the type of instrument used to measure the humidity.
- d) A complete description of the exposure cycle used, including the following information for each light and dark period:
 - 1) the mean and the tolerance limits for the temperature recorded by the black-panel temperature sensor used:

- 2) the mean and the tolerance limits for the relative humidity of the air passing over the test specimens;
- 3) for tests which include a water spray period, report the duration of the water spray and whether the water was sprayed on the exposed face, the back or both surfaces of the specimens; if the total solids of the water used for the spray is greater than 1 μg/g, report the total solids and the silica content;
- 4) for tests where water is condensed on the specimens, report the length of the condensation period;
- 5) the length of each light and dark period.
- 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.
- f) The procedure for test specimen repositioning, if used.
- A description of the radiometer used for measuring the light dosage, if used.
- 7.3 Test results.
- a) A complete description of the test procedure used for measurement of any properties reported.
- b) The results, presented in accordance with ISO 4582, and including:
 - 1) the results from property measurements on the test specimens;
 - 2) the results from property measurements on control specimens;
 - the results from property measurements on unexposed file specimens, if determined;
 - 4) the exposure period (either the time in hours, or the radiant energy in J·m⁻² and the bandpass in which it was measured).
- 9.4 The date of the test.

Annex A

(informative)

Factors that decrease the degree of correlation between accelerated tests using laboratory light sources and actual-use exposures

A.1 Differences between the spectral distribution of the laboratory light source and that of daylight

Exposures at shorter-than-normal wavelengths are often used to obtain faster failure rates in accelerated laboratory exposure tests. For outdoor exposures, the cut-on for short-wavelength UV radiation is generally considered to be about 300 nm. Exposures to UV radiation of wavelengths less than 300 nm may produce degradation reactions which do not occur when the material is used outdoors. If a laboratory light source used in an accelerated test produces UV radiation of wavelengths shorter than that found in the actual-use conditions, the mechanism of degradation and the stability ranking of the materials tested can be dramatically different in the accelerated test.

It may not be necessary to simulate daylight over the entire spectrum, if radiation in a specific region is known to produce the type of degradation of interest in the materials being tested. However, laboratory light sources which have a very strong emission in a narrow band relative to the rest of the ultra-violet or visible spectrum may cause a particular reaction to be favoured relative to others which may be very important. This type of light source may also not produce changes caused in exposures to daylight. Exposures to light sources which only produce ultraviolet radiation may not produce the colour fade caused by visible radiation, and may cause polymer yellowing that is more pronounced than that produced in exposures to daylight.

A.2 Light intensities higher than those experienced in actual-use conditions

Light intensities higher than those experienced in actual-use conditions are typically used in exposures to laboratory light sources in order to accelerate degradation. There are several reasons why the use of abnormally high irradiance can change the mechanism of material degradation relative to the conditions found in an actual-use environment (since some materials are more sensitive to changes in light intensity than others, the use of abnormally high irradiance can alter the stability ranking of the materials):

- a) In exterior exposures, polymers in an excited state caused by absorption of a high-energy photon will typically decay to the ground state before absorbing another high-energy photon. However, in exposures to laboratory light sources producing an abnormally high light flux, the rate of photon absorption is so high that the material will often absorb a high-energy photon when it is still in an excited state.
- b) Free radicals are often formed in materials exposed to ultraviolet light. Reactions leading to degradation occur when the free radicals interact with the material. Free radicals can also recombine with other free radicals in reactions that do not lead to degradation. The high concentration of free radicals formed under high-irradiance conditions results in a greater percentage of recombination due to the close proximity of the free radicals. In this case, degradation is not a linear function of irradiance.
- c) Oxygen diffusion can sometimes become rate-limiting in polymer oxidation processes where abnormally high irradiance (or abnormally high specimen temperatures) are used for test acceleration. This can produce differences in the mechanism of degradation reactions and may cause an abnormal ratio of surface to bulk oxidation, which could result in unnatural colour shifts or physical-property changes.

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A.3 Continuous exposure to light from a laboratory light source without any dark periods

Continuous exposure to light from laboratory light sources is often used in order to achieve accelerated degradation relative to actual-use conditions. However, continuous exposure to light may eliminate critical dark reactions that occur in outdoor exposures or indoor-use conditions where there are regular periods without light.

A.4 Specimen temperatures that are abnormally high relative to actual-use conditions

Temperatures higher than those experienced in actual-use conditions are often used to obtain faster degradation in accelerated laboratory tests. Some plastics are much more susceptible to degradation due to thermal effects than others. For materials that are subject to the same rate and type of photodegradation, exposures at abnormally high temperatures may make a temperature-sensitive material appear to be less durable than a less temperature-sensitive material. In addition, exposures of polymers at temperatures above their glass-transition temperature can dramatically alter the mechanism of degradation and the stability ranking compared to exposures conducted at a temperature below the glass-transition temperature. The black-panel temperature used in an accelerated laboratory test should be maintained in a reasonable range, which is usually no higher than the maximum observed for the black panel in actual-use conditions.

A.5 Exposure conditions that produce unrealistically large temperature differences between light- and dark-coloured specimens

Some laboratory light sources produce large amounts of infrared radiation. In order to prevent overheating of the specimens, the infrared radiation can be reduced by using infrared-absorbing or -reflecting filters, or by passing large amounts of air through the specimen chamber to cool the specimens. If measures to control the amount of infrared radiation reaching the specimens being exposed are not sufficient, the temperature differences between light- and dark-coloured specimens of the same material can be much larger than would be seen in natural exposures.

Some laboratory light sources produce very little infrared radiation. When these types of laboratory light source are used, the difference between dark- and light-coloured specimens may be much less than that found in outdoor exposures.

A.6 Temperature-cycling conditions that are different from those found in actual-use

Abnormally high temperature-cycling frequencies can produce mechanically induced cracking or other types of degradation not seen in exposures under use conditions. Exposure devices which spray specimens with water when the light source is on can produce an abnormally rapid change in temperature that may also produce cracking not produced in the use environment.

A.7 Unrealistically high or low levels of moisture

Moisture is very important in producing degradation of many polymers. If the amount of moisture, or the way in which specimens are exposed to the effects of moisture in an accelerated laboratory test, differs from that in the actual-use environment, the mechanism and rate of degradation may be very different. This can have a significant effect on the stability ranking of the materials.

A.8 Absence of biological agents and pollutants

Plastic materials exposed in warm, wet locations are often subject to significant growth of biological agents such as fungi, bacteria and algae. Pollutants present in many exterior environments have a significant effect on the mechanism and rate of degradation of some plastics. If these effects are not included in an accelerated laboratory exposure test, the mechanism and stability ranking of the materials may be significantly different from that found in exterior exposures.

Annex B

(normative)

Procedures for measuring the irradiance uniformity in the specimen exposure area

B.1 In devices that use a drum to hold the specimens and rotate them a round a light source, measure irradiance at a position in the specimen drum that is closest to the light source (position A in Figure B.1) and at two positions within the specimen drum that are farthest from the light source (position B) in Figure B.1. The relationship between the irradiance at position B relative to the irradiance at position A shall be as follows:

 $E_{\rm B} \ge 0.7 E_{\rm A}$

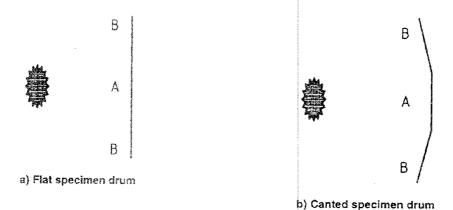


Figure B.1 — Measurement of irradiance in devices using a rotating specimen drum

B.2 In devices where specimens are positioned in a flat plane in front of a light source, measure irradiance at a position in the specimen plane that is closest to the light source (position X in Figure B.2) and in two opposite corners of the plane where test specimens are placed (position Y in Figure B.2). The relationship between the irradiance at position Y relative to the irradiance at position X shall be as follows:

 $E_{\rm Y} \ge 0.7 E_{\rm X}$

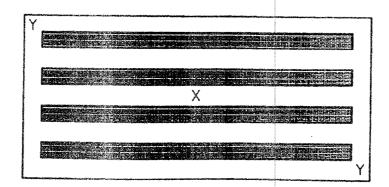
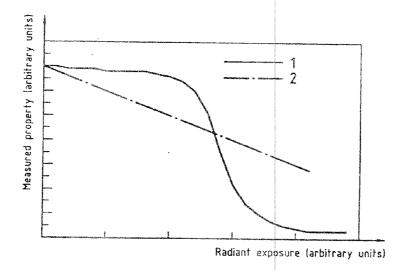


Figure B.2 — Measurement of irradiance uniformity in devices with a flat specimen plane (shaded areas indicate light sources)

B.3 If the design of the device is such that the maximum irradiance may not be at the centre of the exposure area, or that the minimum irradiance may not be at the position farthest from the centre, the actual maximum irradiance shall be used for $E_{\rm A}$ or $E_{\rm X}$ and the actual minimum irradiance shall be used for $E_{\rm B}$ or $E_{\rm Y}$ in the equations in B.1 and B.2. Additional measurements of the irradiance at other positions within the exposure area may also be made. In all cases, however, the irradiance measured at these positions shall be at least 70 % of the maximum irradiance.

B.4 As an alternative to irradiance measurements, uniformity of irradiance may be determined by use of reference materials. The change in the characteristic property of the reference radiant exposure (preferably linear) and should preferably not show property as a function of radiant exposure. Figure B.3 is a typical plot of measured property as a function of radiant exposure for acceptable and unacceptable reference materials. When reference materials are used, all specimens shall be from the same lot. Expose reference material specimens at the centre of the exposure area and at positions there is a measurable change in the characteristic property being monitored. The change in the measured property of the reference material at positions farthest from the centre shall be at least 70 % of the change measured on the specimen exposed at the centre.

NOTE Actual measurements of irradiance are preferred over the use of reference materials because differences in property change between reference material specimens exposed at the extremes of the exposure area and those exposed at the centre may be significantly affected by differences in temperature and/or moisture conditions as well as differences in irradiance.



Key

- 1 Unacceptable
- 2 Acceptable

Figure B.3 — Typical plot of measured property for a reference material showing a linear change with radiant exposure and for a material that shows an induction period before any measurable property change